VOL. XXXVI 1919

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TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

VOLUME XXXVI

THIRTY-SIXTH GENERAL MEETING

CHICAGO, ILLINOIS

SEPTEMBER 23, 24, 25 AND 26, 1919

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VOLUME XXXVI 1919.

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES AND RECORD OF THE THIRTY-SIXTH GENERAL MEETING OF THE SOCIETY, HELD AT THE CONGRESS HOTEL, CHICAGO, SEPTEMBER 23, 24, 25 AND 26, 1919.

Number of members registered, 242; number of guests registered, 136; total, 378.

PROCEEDINGS OF TUESDAY, SEPTEMBER 23, 1919.

The all-day excursion planned for this day to Gary, Indiana, and trip through the Indiana Steel Company's plant, had to be abandoned on account of a strike in the steel industries. At 2 o'clock P. M. a joint technical session was held with the American Institute of Mining and Metallurgical Engineers on the subject of iron and steel. This session was continued at 8 P. M. at the Congress Hotel.

PROCEEDINGS OF WEDNESDAY, SEPTEMBER 24.

Registration, which had already been started at headquarters, Florentine Room, Congress Hotel, at 9 A. M. the previous day, was resumed at 9 A. M. at the same place.

The meeting was called to order by President W. D. Bancroft at 10 A. M. The first business on the program was the presentation of a final report by the committee on the Algebraic Signs of Potentials. This report, together with ensuing discussion, is appended to these Proceedings.

Reading and discussion of papers was taken up, and papers by the following considered: M. A. Hunter and J. W. Bacon,

A. J. Helfrecht, N. K. Chaney, Louis Kahlenberg and J. A. Montgomery (2 papers), T. S. Fuller. These papers and their discussions are printed in full in these Transactions.

After recess for luncheon a joint technical session with the American Institute of Mining and Metallurgical Engineers was called to order in the auditorium at the Fifth National Exposition of Chemical Industries. This Society submitted papers by the following: F. A. J. FitzGerald (2 papers), A. M. Clark and H. G. Weidenthal. These papers and their discussions are printed in full in these Transactions.

The evening was devoted to seeing electric furnace exhibits at the Exposition.

PROCEEDINGS OF THURSDAY, SEPTEMBER 25.

At 10 A. M. a joint technical session was held with the A. I. M. & M. E. at the Congress Hotel; the subject was a Symposium on Pyrometry. The record of this session has been printed in full by the A. I. M. & M. E. in its Transactions. After luncheon this joint session was continued and concluded. At 8 P. M. a complimentary Smoker, tendered by the Local Committee, was held in the Florentine Room of the Congress Hotel. In addition to various other entertaining features, the Edison Symphony Orchestra and the Commonwealth Edison Glee Club contributed to make the evening highly enjoyable.

PROCEEDINGS OF FRIDAY, SEPTEMBER 26.

At 10 A. M. a large assembly gathered in the auditorium of the Exposition for the Symposium on Catalysis, in charge of Prof. H. S. Taylor. The papers presented were by the following: W. D. Bancroft, H. S. Taylor, W. J. Huff, F. C. Zeisberg and E. K. Rideal. These papers and their discussions are printed in full in these Transactions. After luncheon at the Exposition the Symposium on Catalysis was continued and concluded, papers by the following being presented and discussed: William Blum; William Blum, F. J. Liscomb, Zalia Jenks and W. E. Baily; and C. J. Thatcher. These papers, together with their discussions, are printed in full in these Transactions. The next feature of the afternoon was a demonstration of the manufacture of fluor-

ine by Prof. F. C. Mathers. This process of preparing fluorine has been fully described in our Transactions.[1] Dr. Carl Hering then offered the following:

RESOLUTION OF THANKS.

The American Electrochemical Society, closing its 36th General Meeting, in Chicago, wishes to place on record its obligations to those who have contributed in various ways to the success of the meeting:

To the United States Steel Corporation, for its invitation to visit the Gary Steel Plant; the carrying out of which plan was prevented by circumstances beyond its control.

To the management of the Fifth National Exposition of Chemical Industries, for its hospitality in connection with various sessions of the meeting.

To the management of the Congress Hotel, for accommodations as hotel headquarters and for holding other sessions.

To the Edison Symphony Orchestra and the Commonwealth Edison Glee Club, together with the committee in charge of the complimentary smoker and entertainment, for a highly interesting and agreeable evening's relaxation.

To the Ladies' Committee, for their very capable reception and entertainment of visiting ladies.

To the Electric Furnace Co., the Detroit Electric Furnace Co., and the Shawinigan Water and Power Co., for their exhibitions of motion pictures illustrating electrochemical plants.

To the General Local Committee, for its efficient assistance in arranging the many details of the meeting.

The above motion was unanimously adopted.

FINAL REPORT OF THE COMMITTEE ON THE ALGEBRAIC SIGNS OF POTENTIALS.

The Committee recommends that the signs given to the potentials of electrodes be those which represent their electric charges with reference to the solutions. An element like zinc, which becomes negatively charged with respect to the solution, is therefore

[1]Trans. Am. Electrochem. Soc., 1919, **35**, 335.

given a negative sign, and the sign of the calomel electrode then is plus. In charging and discharging a storage battery the current is reversed but the polarity remains the same.

This recommendation is in agreement with the more generally adopted international practice.

The assumed absolute zero of potential or zero of reference should accompany the data.

(Signed) CARL HERING, Chairman,
FRANCIS C. FRARY,
M. DE K. THOMPSON.

New York City, April, 1919.

Minority Report.

I dissent from the above recommendation for reasons set forth in Vol. **34**, p. 189 *et seq.*, of the Transactions of this Society, and in controversion of the statement that "This recommendation is in agreement with the more generally adopted international practice," I append to the list there given the following authorities who are opposed to the use of the signs of potentials as above recommended:

H. C. Jones. Outlines of Electrochemistry, p. 96. (Published by Van Nostrand, 1901.)) Elements of Physical Chemistry, p. 408. (Macmillan, 1902.)

Arrhenius. Text Book of Electrochemistry, p. 237. (Longmans, Green & Co., 1902.)

M. de K. Thompson. Applied Electrochemistry, p. 22. (Macmillan, 1911.) Nernst. Theoretical Chemistry, p. 764. (Macmillan, 1911.)

A. A. Noyes & Sherrill. General Principles of Chemistry, p. 164. (1917.)

Bureau of Standards. Rawdon, Grossman, and Finn. Chem. and Met. Eng., Vol. **20**, p. 458. (1919.)

(Signed) OLIVER P. WATTS.

Madison, Wis., Sept. 20, 1919.

Appendix to the Final Report of the Committee on the Algebraic Signs of Potentials.

By CARL HERING.

In general, a plus sign implies the adjective "more" or "higher" and a negative sign "less" or "lower." Hence in the present case a plus sign implies a relatively greater activity and a negative sign a relatively less activity. One of the objections which has been

made to giving metals like zinc the negative sign, is that these metals are in general the ones which are the more active from the chemical standpoint, and their chemical potential or activity would therefore naturally be given the plus sign. There is therefore an apparent disagreement between the chemical activity and the electrical activity, when as a matter of fact chemical and electrical activities in general go together.

It is now known, however, that it is the negative electrons which are the active ones and that therefore the flow of an electric current is in fact in the opposite direction to what has long been and is still being conventionally assumed. A greater charge of negative electrons therefore represents a greater electrical activity. Hence, by giving the negative sign of potential to metals like zinc, which are in general the more active from a chemical standpoint, it brings the chemical activity and the electrical activity into agreement with each other.

Another source of confusion is that the direction of flow of the current in the external circuit when referred to the poles or terminals, is necessarily the opposite to that in the interior of the cell, dynamo or other source. That is, if it is assumed conventionally to flow from the positive to the negative pole in the external circuit, it necessarily flows from the negative to the positive pole in the interior of the cell. Hence in connection with the signs of the potentials of electrodes it makes a difference whether one is referring to the external or to the internal part of an electric circuit. Referred to the external circuit the chemically more active metals like zinc are generally negative, and are the ones to be connected to the negative pole of a voltmeter.

DISCUSSION.

F. C. FRARY[1]: Inasmuch as Mr. Watts and I started this argument and we both have had a great deal to say, I have taken the liberty of reducing my remarks to writing, so as to try to condense them as much as possible.

In his reply to my discussion of his minority report on this subject, Dr. Watts persists in misunderstanding my remarks.[2]

[1] Director of Research, Aluminum Co. of America, New Kensington, Pa.

[2] Trans. Am. Electrochem. Soc., 1918, 34, 200.

He asks me to cite the reference where I think he claims that the current does not flow from the zinc to the copper in the voltaic cell. If he will refer to his own paper which I was discussing, he will find his own statement to which I referred, when I stated that he objected to our saying that the current flows from the negative pole (zinc) to the positive pole (copper), as follows:

"If, however, zinc be considered as electro-negative and copper as positive, there will no longer be harmony in the use of signs in the voltaic and electrolytic cells, but we will be compelled to say that current flows in the electrolyte from the positive to the negative electrode in the electrolytic cell, but from the negative to the positive electrode in the voltaic cell—in the latter case outraging our fundamental conception of the terms positive and negative as used everywhere else in science and industry." Dr. Watts chooses to ignore the very obvious meaning of my remarks in order to accuse me of a misquotation. The flow of the current from the negative electrode to the positive electrode is exactly what he objected to.

Now Dr. Watts admits in the same paper that the zinc is actually negative to the solution and that he would attach it to the negative terminal of his voltmeter. But he objects to our calling it a negative electrode, and calls it an anode because the current (positive charges) flows from it into the solution. My contention is that we do not care about the direction of flow in the solution: as Dr. Watts is doubtless aware the term "anode" in its practical everyday use is limited to electrolytic cells and designates the electrode to which the plus terminal of the voltmeter is connected. In order to make clear to him the difficulties of attempting to define the sign of the electrode by the direction of flow of the positive charges in solution I used the analogy of the motor and dynamo, but Dr. Watts by relying on his definition of anode attempts to show that the case of passing a current through a solution is "not at all a parallel case." In order to make it perfectly clear even to Dr. Watts I will take his own case of zinc and copper immersed in an electrolyte.

Suppose we have a battery composed of a zinc strip and a copper strip separated by a porous diaphragm, immersed in dilute sulphuric acid and connected to a shunt-field type toy motor, with a voltmeter connected in the usual way across the line. When

the circuit is closed, the motor runs and the zinc dissolves. If we have connected the negative terminal of our voltmeter to the zinc and the positive to the copper, the voltmeter will register about one volt. Power is being generated by the cell and consumed by the motor; the current (positive charges) in the motor armature flows from the positive brush to the negative, and in the solution it flows from the zinc to the copper. Dr. Watts says that this makes the zinc the anode and that therefore if I should measure its potential against the solution I ought to give the result a positive sign. My contention is that the zinc is actually negative in respect to the solution, and that this potential should be given a negative sign. I do not call it an anode, because I consider this term as applicable only to cells where work is being done on the cell and not by it, but am content to designate it as the negative electrode, because it actually is negative with respect both to the solution and the other electrode, as is shown by the fact that even Dr. Watts admits he must connect the negative terminal of his voltmeter to it.

Now suppose the battery has been running some time, and the acid has been largely consumed in the formation of zinc sulphate. The motor still runs and the current flows throughout the circuit in the same direction as before. Now I put a belt on the pulley of the motor and proceed to apply power to it, revolving it in the same direction as before but at a higher speed. My motor now becomes a dynamo, turning mechanical work into electrical energy. The current in the armature now reverses and flows (positive charges) from the negative brush to the positive one, out along the line to the copper electrode and then back through the solution to the zinc electrode and to the negative brush. The fact that I have not changed the polarity of either the dynamo or the cell is shown by the voltmeter, which, without changing its connections, now registers a higher voltage than it did, but shows the zinc to be still negative with respect to the copper.

But in the solution I have reversed the actual direction of the current: it now flows from the copper to the zinc. Dr. Watts has defined the anode as the electrode where the positive charges enter the solution: this is now the copper electrode, and according to his ideas its potential measured against the solution should be given a positive sign; contrariwise the potential of the zinc against

the solution would have to be given a negative sign, as far as I can see. I am in agreement with him here, because I find that the zinc still is actually negative to the solution; in fact it is actually a little more negative than it was before. To me the difference between its state now and in the first case is merely one of degree: I have done work upon it and forced it to become more negative than it wanted to be, with the result that instead of sending out its (positive) ions into solution it attracts them again to itself, and more or less zinc is deposited upon the zinc electrode.

Actually, if I was to measure this difference of potential between the electrode and the solution with the usual apparatus (calomel half cell and potentiometer), I would find that the difference in potential between the electrode and the solution was still in the same sense (*i. e.*, I would not have to reverse my potentiometer connections) and only slightly greater numerically than it was before. If I chose to apply my power to the machine very gradually and follow the potential changes of the electrode I could show that there was no break, or change of sign of the potential between the electrode and the solution: there would only be a gradual increase in the numerical value of the difference of potential. According to Dr. Watts' definitions, the zinc was at first anode, and its electrode potential must be given a positive sign; when the current reversed, the sign reversed and the zinc became cathode so that its potential could properly be given the negative sign, which I contend it should have had all the time, in order to be consistent with the facts.

Dr. Watts states (*loc. cit.*, p. 201): "The motor and dynamo, which Mr. Frary cites as being 'exactly analogous' to the relations between the electrolytic and voltaic cells, is not a parallel case; for, as Mr. Frary himself points out, when a motor acts as a dynamo, 'the armature current is reversed,' but in both the electrolytic and voltaic cells the direction of flow of current is always from anode to cathode. Therefore whatever sign is used for the anode of the electrolytic cell should be used for that of the voltaic cell also, if we are to be consistent."

Dr. Watts forgets that I am speaking in terms of facts and he in terms of his own assumed definitions. I implied that the actual direction of the current in the cell could be reversed by putting energy into it instead of taking it out, exactly as is the

case of the dynamo-motor combination, and as I have shown it to be in the case described above. This actual reversal doesn't matter to Dr. Watts because he just changes the labels on his electrodes and says the current still flows from anode to cathode. Of course it does, assuming his definitions, but that doesn't in the least alter the fact that the actual direction of the current in the cell has been reversed, just as it was in the motor and dynamo.

Dr. Watts is disturbed about the possibility that if we give the electrode potentials the signs advocated by the majority of the committee "we shall still fail to express the truth regarding the potentials of metals, for the latest theory of the nature of electricity leads us to believe the zinc electrode is really at a higher potential than the electrolyte, and consequently the plus and not the minus sign indicates its potential." He is still begging the question by the assumption that what we express by the plus and the minus signs in electricity is a higher or lower potential, whatever that may mean, while what we really mean in any branch of the subject is that a given point to which we assign a plus potential (be it a pole of an electro-static machine, a condenser plate, or a dynamo terminal) has been electrically displaced from a neutral position so that it contains more electrical charges of the kind which we have arbitrarily designated as plus than it has of the kind which we designated as minus. Similarly a negative point has more negative than positive charges upon it. When we speak of an electrode or wire as having a high or higher potential we simply mean that there is a great difference of potential between it and its surroundings, usually referring to the ground as neutral, but we speak of it as a high potential irrespective of whether it happens to be charged with plus or minus electricity: it is only the density of the charge which is considered and not its character. Consequently if we decide that negative charges are the only kind which exist, we do not get into any trouble because the negative electrode potential of zinc simply means that it has an excess of these charges and hence has taken their sign, while an electrode which had a positive electrode potential would simply be deficient in negative charges, and therefore take on the electrical state which we designate as plus or positive.

To make my point perfectly clear I will go still further and cite the case of a cell having a zinc electrode and a magnesium elec-

trode immersed in the same electrolyte, which we may assume again to be sulphuric acid. If the two electrodes are connected by a wire, the current (positive charges) will flow along the wire from the zinc to the magnesium, and in the solution from the magnesium to the zinc. Now, according to Dr. Watts' definition, the magnesium is here the anode, since it discharges its positively charged ions into solution, and consequently the zinc must be cathode. The electrode potential of the magnesium would then, according to his definition, be given a positive sign, while that of the zinc could not be, since the current does not enter the solution there, and therefore it would have to be given a negative one. According to Dr. Watts' own table there will show an E. M. F. of about 0.73 volt between these two electrodes. But if I should actually measure these electrode potentials, according to the same table, I would find the potential between the magnesium and the solution to be 1.23 volts while that between the zinc and the solution is 0.50 volt.

If I have given the magnesium potential a positive sign on the ground that the magnesium is anode, and the zinc a negative sign on the ground that it is cathode, I must wind up with the calculation that the E. M. F. of the cell should be 1.73 volts instead of 0.73. But of course when I actually measure these E. M. F.'s, I see at once from my potentiometer that the difference in potential between the electrode and the solution has the same sign in both cases; *i. e.*, both electrodes are trying to send their positively charged ions into solution. But if I couple them together into a cell, only one of them can do so, and the magnesium being the stronger might be said to overpower the zinc and have things its own way.

Dr. Watts at once replies that the magnesium being stronger then takes the positive sign. But how about the zinc? I have been unable to find any place where he actually says that the cathode must always have the negative sign, but he certainly indicates this when he says (p. 200): "To this designation of the anode as p o s i t i v e in those particular combinations of two electrodes and an electrolyte which function as an electrolytic cell, but as n e g a t i v e in similar combinations of electrodes and electrolyte that constitute a primary cell, I emphatically object." If the anode must always be positive we can only draw

the conclusion that the controlling factor in the sign of the electrode must be (assuming Dr. Watts' definition of anode) whether or not positive charges flow from it into the electrolyte. There can be no half way about it. Either we have to define the sign of the potential of the electrode by the way in which the current happens to flow in the electrolyte, or else we have to do it independently of the flow of the current in particular cases, but dependent only on the actual difference in sign of the electrode and solution.

I have tried to show that definition on the basis of current flow in the electrolyte is impractical. I personally see no inconsistency in the statement that under some circumstances positive charges enter the electrolyte from the negative electrode, and that under other circumstances negative charges enter from the same point. It is no more inconsistent than to use electricity to produce heat in one machine and cold in another.

Dr. Watts and Dr. Richards speak as though the designation I have advocated was a radical change which would introduce confusion everywhere, although Dr. Watts admits that the confusion already exists. As a matter of fact, this method of giving the electrode potential the actual sign of the potential of the electrode as referred to either the solution or an assumed neutral electrode which would not send any ions into the solution, has been adopted abroad for many years (see Bancroft, these Transactions, 1918, **33**, 80), and our use of it will tend to unify and not diversify the world's practice. Confusion in the theory of the voltaic cell is easily avoided by restricting the use of the terms anode and cathode to electrolytic cells where work is being done by current introduced from outside, and the zinc in a voltaic cell then becomes simply the negative electrode, from which negative electricity flows just as it flows from the negative terminal of a dynamo, and to which we connect as a matter of course the negative terminal of the voltmeter. If we allow the battery to supply energy to an outside circuit, the electric charges flow from their respective electrodes; while if we do work upon it, as in charging the storage battery, the electric charges flow toward their respective electrodes. The thing is simple, easily understood, and in accordance with the facts.

While I believe every one of the four members of the majority

of the committee had a different method of explaining or viewing this matter, we all came to the same conclusion, that we should actually give to an electrode potential the sign which designated its character or the character of its charge as it referred to the solution or to an assumed neutral electrode which is supposed not to send any ions into the solution.

W. D. BANCROFT[3]: The more I hear of the reasons for that majority report, the less certain I feel of my ground. Possibly our confidence in it could be restored by hearing arguments on the other side.

O. P. WATTS[4]: Mr. Chairman, I came in in the midst of this rather lengthy discussion by Mr. Frary, and was unable to get the whole of it; in fact it was so lengthy and comprised so many points that I was unable to grasp all that was given while I was present. For that reason I will not attempt to answer his discussion as a whole at this time. I note that Mr. Frary would limit the use of the term anode to the electrolytic cell. I would like to know by what authority. The term anode has been applied to the primary cell from the early days of electrochemistry, and as far as I have observed, no one has since said it should not be so applied until Mr. Frary said so today. He spoke of my "assumed definition of anode." I had not assumed any definition of an anode. That definition was given us by Faraday and it is beyond any assumption on the part of either Mr. Frary or me. I would like to ask Mr. Frary in terms of his views of their potentials, how he would express a case of corrosion of this kind: in the literature we often see mention of the acceleration of corrosion of iron produced by its contact with copper, and it is commonly said to occur because the copper is negative to the iron. I would like to ask Mr. Frary how he would explain the accelerated corrosion of a piece of iron produced by contact of copper according to his electrochemical series, in which he says copper is positive and iron negative?

F. C. FRARY: I would simply say that the iron has a greater solution tension than the copper and we have there a short circuited electrolytic cell in which iron functions as the negative electrode and the copper as the positive electrode.

[3] Professor of Physical Chemistry, Cornell University.

[4] Associate Prof. of Chemical Engineering, University of Wisconsin.

O. P. WATTS: But your solution has not been changed by the contact of the copper with it.

F. C. FRARY: No, but it is very well known that the reason why the combination of iron and copper functions that way is because the over-voltage of hydrogen on copper is low enough so we could evolve the gaseous hydrogen there, whereas we could not on the iron. Normally the iron would be protected by the over-voltage of hydrogen.

O. P. WATTS (*Communicated*): It would lengthen too much a controversy that has already been prolonged unduly, to take up every point in Mr. Frary's ten-page discussion. His argument seems to be based on the assumption that I and others who contend against his reversing signs in the electrochemical series, consider that any metal which can be made to serve as cathode in a voltaic or electrolytic cell must be given a negative sign in the electrochemical series. I have never so thought or contended. For all practical purposes the position of the zero point in the electrochemical series has no greater significance than has the zero on a thermometer scale. Just as a particular body may have its temperature recorded as being above or below zero, depending on whether a Fahrenheit or Centigrade thermometer be used to measure the temperature, so the potentials of certain metals are either positive or negative, according to the assumed potential of the reference electrode against which they are measured, and the electrolyte with which they make contact.

If a voltaic cell be made by dipping any two metals into an electrolyte, the one that is higher will function as anode, no matter whether the potential of either or both lie above or below the zero point of the potential scale—just as between two bodies of unequal temperature the flow of heat is f r o m the warmer of the two, regardless of whether the temperature of one or both of them is below zero on the scale of the particular thermometer used for measuring their temperature.

This principle was clearly stated in the first paragraph (p. 192) of my paper in Vol. **34**, to which Mr. Frary referred at the beginning of his discussion. To assume that copper, for example, must always be cathode (*i. e.*, that current will flow to it) because its potential in an electrochemical series is negative, is like saying

that a brick whose temperature is 5 degrees below zero (— 5° C.) must always absorb heat from any body with which it may be put in contact, regardless of the temperature of the other body.

J. W. RICHARDS[5]: It strikes me, gentlemen, that we have two different views on the subject, which I will call the chemical and the physical. I have always looked at it primarily from the chemical side, and that has led me to favor Dr. Watts' designation; I believe that view is absolutely correct from a chemical standpoint. The other method is the physical, and has been developed by the electrician or electrical engineer; he says that in order to make these signs conform with the conventional signs which are used, they should be the reverse of the chemical, and the chemically active metal should be called negative: the more active they are, the more strongly negative they are. I hope I can make this clear. It seems to me that if the physicists had started out by calling the direction of the current the opposite direction from the conventional one, the direction in which they now think the current goes, there would be no confusion, and we would have no discussion. The signs then asked for by the electrical engineers would conform and agree with those which the chemists insist are correct. Now the physicists admit that their conventional direction of the current was wrong, and they ask that the signs of the potential which the chemist uses be also made wrong in order to agree with their conventional method. Well, it is a fair question which should be changed. The physicist says it is impossible, at the present time, to change the conventional signs which are now used by him, and therefore, assuming that they must be retained, the chemist should invert his signs in order to correspond with the conventional signs used by the physicist. It seems to me it is a matter of convenience; which is the best, for the chemist to persist in his signs which I think the physicist now admits are chemically correct, although opposed to the conventional signs, or shall the chemist accept the conventional signs which are admittedly wrong, and say, "Well, let us call our chemically active metals chemically negative, and the more chemically active they are, the more negative they shall be called." I think it is a matter of weighing the advantages and disadvantages. Who shall give way? Which is the most prac-

[5] Professor of Metallurgy, Lehigh University.

ticable, to invert the signs chemically or invert the signs which are used in engineering? The easier thing to do, the path of least resistance, is to keep the conventional signs and change the chemical signs to correspond.

CARL HERING[6]: There is another illustration of the point made by Dr. Richards. Many years ago our forefathers said the sun rose. Since then we found that the sun does not rise but the earth turns around; yet in our daily papers and I believe even in the Nautical Almanac, it is still said that the sun rises, and there is no confusion caused by it.

COLIN G. FINK[7]: I move that the majority report be adopted by the Society. This involves that all publications by the Society will conform with this majority report, and any papers submitted to the Society having potential signs that do not comply with this majority report, be carefully edited by our Publication Committee.

The resolution was seconded and adopted.

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Williams, H. M.

Hartford—
Gilligan, Frank P.
Palmer, W. R.
Peiler, Karl E.
Pettee, C. L. W.
Willson, E. L.

Meriden—
Fitz, Arthur E.
Thomas, Geo. E.

New Britain—
Andrews, J. C.
Hoot, D. W.

New Haven—
Bradley, W. M.
Brown, W. T. K.
Gravely, J. S.
Hibbert, Harold
Johnston, John
Scott, Chas. F.

Seymour—
Dana, A. S.

Stamford—
Ferguson, C. N.
Getman, F. H.
Given, G. C.

Stepney Depot—
Wheeler, Fred B.

CONNECTICUT—Continued

Wallingford—
Blanchard, H. J.
Canby, Robt. C.
Marsh, R. J.

Waterbury—
Bassett, W. H.
Bedworth, H. A.
Bennett, M. H.
Bradley, J. C.
Bristol, W. H.
Brockway, J. P.
Cowperthwait, A. D.
Hogaboom, G. B.
Price, Wm. B.
Richardson, H. K.
Sperry, R. S.
Taylor, F. D.
Thompson, H. L.
Weiser, F. S.

Westport—
Curtis, F. J.

DELAWARE

Henry Clay—
Bradshaw, H.

Seaford—
Morse, W. S.

Wilmington—
Buchanan, A. E., Jr.
DuPont, Irenee
DuPont, Lammot
DuPont, Pierre S.
Fox, H. W.
Hitch, A. R.
Holmes, F. B.
Humiston, Burr
Lee, I. E.
Liljenroth, F. G.
Lloyd, S. C.
MacGregor, F. S.
Norman, Geo. M.
Reese, C. L.
Sparre, Fin
Symmes, E. M.
Teeple, O. J., Jr.
Whitten, Wm. M., Jr.
Williams, Roger
Yount, A. S.
Zeisberg, F. C.

DISTRICT OF COLUMBIA

Washington—
Adams, Quinton
Bancroft, W. D.
Bate, H. A.
Blum, William
Casey, G. L.
Coggeshall, G. W.
Cottrell, F. G.
Cushman, A. S.
Dewey, F. P.
Dodson, F. W.
Frankforter, G. B.
Freeman, J. R., Jr.
Gaillard, D. St. Pierre
Gardner, H. A.
Gilchrist, Raleigh
Gottlieb, M. B.
Holton, F. A.
Hopkins, N. M.
Jones, Grinnell
Kinney, S. P.

DISTRICT OF COLUMBIA—Cont.

Washington—Continued
Landolt, P. E.
Lloyd, M. G.
Lyon, D. A.
Marshall, S. B.
Massey, M. F.
Moody, Herbert R.
Munch, J. C.
Nash, P. M.
Naylon, J. T.
Parsons, C. L.
Pleiss, Paul
Reed, A. C.
Rich, Wm. J.
Richardson, C. N.
Roberts, C. H. M.
Russell, Bert
Short, Frank
Townsend, C. P.
Turrentine, J. W.
Watson, P. B.
Witherspoon, T. A.

FLORIDA

Beresford—
Corson, E. F.

Ocala—
Martin, R. M.

Panasoffkee—
Tanner, W. L.

GEORGIA

Atlanta—
Adsit, C. G.
Maynard, T. P.
Wood, H. P.

IDAHO

Kellogg—
Bottinelli, M. J.

Pioneerville—
Koering, B. K.

Wallace—
Day, J. J.

ILLINOIS

Belleville—
Malinovszky, Andrew

Berwyn—
Crabbe, Arthur

Chicago—
Arthur, Paul
Baker, Chas. E.
Baker, R. E.
Booth, W. K.
Brady, W.
Brunt, H. H.
Coffelt, O. T.
Condit, B. C.
Converse, W. A.
Cummins, A. B.
deBeers, F. M.
Dengler, F. P.
Egloff, Gustav
Ericson, E. J.
Eshbaugh, C. H.
Gaylord, C. H.
Golick, T. F.
Gottschalk, V. H.
Greenlee, W. B.
Gudeman, E.
Harkins, W. D.
Harkness, W. E.

ILLINOIS—Continued

Chicago—Continued
Herz, Alfred
Hollis, H. L.
Hosford, Wm. F.
Hoskins, W.
Hunter, J. V.
Jenista, Geo. J.
Johnson, A. R.
Jones, A. C.
Jones, C. H.
Kawin, Chas. C.
Kern, P. E.
Klemm, H. F.
Krejci, M. W.
Lamoureux, Ernest
Liscomb, F. J.
Lloyd, E. W.
Lundgren, H.
Lunn, E.
Lyman, J.
McCormack, H.
McCoy, H. N.
McMahon, G. F.
Mohn, Arnold
Morrison, M.
Reymond, P. LeG.
Schott, J. E.
Servis, O. E.
Slough, F. M.
Smythe, E. H.
St. John, H. M.
Stoll, C. G.
Summers, L. L.
Thordarsen, C. H.
Trantin, Jacob, Jr.
Valentin, J. E. C.
Ward, Henry L.
Weightman, H. E.
Weith, A. J.
Wendt, G. L.
Wilcox, W. G.
Winslow, F. E.
Wurster, O. H.
Zimmerman, J. G.

Depue—
Peters, M. F.

East St. Louis—
Junkins, J. R.
Pritz, W. B.

Edison Park—
Gilbertson, H. A.

Elgin—
Hatch, I.

Evanston—
Bauer, Wm. Chas.
Bragg, E. B.
Langdon, S. C.

Highland Park—
Pfanstiehl, Carl

Joliet—
Meaker, G. L.

La Salle—
Carus, Edw. H.
Ede, Joseph A.

Peoria—
Hight, E. S.
Schueler, J. L.

Peru—
Edwards, J. B., Jr.

River Forest—
Jones, Geo. H.

ILLINOIS—Continued

Rockford—
Readette, John

Urbana—
Dietrichson, J. G.
Palmer, C. W.
Parr, S. W.
Reedy, J. H.

Winthrop Harbor—
Aiken, R. H.

INDIANA

Bloomington—
Brown, O. W.
Burlage, H. M.
Mathers, F. C.

Elkhart—
Turnock, E. H., Jr.

Hammond—
Wright, L. R.
Young, J. C.

Indianapolis—
Atkinson, F. C.
Naiden, J. H.
Schildhauer, Edw.
Smith, T. W.

Kokomo—
Arthur, Walter
Udy, M. J.
Wissler, W. A.

Lafayette—
Duncan, T.

Marion—
Case, A. E.

IOWA

Ames—
Towle, N. L.
Wright, H. V.

Bettendorf—
Bretz, J. A.

Burlington—
Muren, A. L.

Cedar Rapids—
Drabelle, J. M.

Centerville—
Peck, E. L.

Iowa City—
Pearce, J. N.

Keokuk—
Davis, H. N.
Knight, F. P. H.
Schleeder, L. B.
Scholl, G. D.
Sheean, J. L.
Weissenburger, G. E.
Wettstein, T. F.
Wilcox, N. T.

KANSAS

Kinsley—
Parkhurst, I. P.

KENTUCKY

Louisville—
Ruby, C. E.

Marion—
Reed, A. H.

Paducah—
Nichols, A. S.

LOUISIANA

Alexandria—
McConnell, J. J.

New Orleans—
Freas, Raymond
Williamson, C. S., Jr.
Winship, W. E.

MAINE

Auburn—
Adams, E. W.
Higgins, C. H.

Augusta—
Wyman, W. S.

Biddeford—
Smith, E. S. C.

Brunswick—
Cram, M. P.

Cumberland Mills—
Gabriel, G. A.

Great Works—
Larchar, A. B.

Lewiston—
Anderson, W. S.
Andrews, A. B.
Robinson, A.

Portland—
Allen, H. I.
Lincoln, E. S.

Rumford—
Lowe, R. E.

Waterville—
Nivison, Robt.

MARYLAND

Annapolis—
McAdam, D. J., Jr.
Webert, L. P.

Baltimore—
Barclay, E. H.
Beck, Wm. H.
Boynton, W. H.
Browne, A. L.
Bryan, J. K.
Kenway, I. F. L.
Lovelace, B. F.
Miller, E. B.
Mitchell, G. K.
Moore, W. C.
Peirce, W. H.
Rouse, E. W., Jr.
Sisco, F. T.
Stone, W. H.
Thompson, M. R.
Tillman, R. H.
Warth, A. H.
Winter, E. J.
Wise, J. B.

Chevy Chase—
Schramm, Edw.

Colgate—
Aldrich, C. H.

Cumberland—
McKaig, W. W.

Emmitsburg—
Rauth, J. W.

Indian Head—
Wagner, A. A.

Luke—
Pedder, John

MARYLAND—Continued

Mount Savage—
Ramsay, Andrew

Rowland Park
Thomas, C. C.

MASSACHUSETTS

Arlington—
Reed, L. N.

Boston—
Atwood, F. C.
Barker, E. R.
Buchanan, L. B.
Campbell, J.
Clapp, E. H.
Comstock, D. F.
Davis, G. S.
Eustis, A. H.
Harrington, R. F.
Hobby, A. K.
Howard, W. D. M.
Hyde, A. T.
Kao, T.
Kniffin, L. M.
Pratt, F. S.
Schenck, Garrett, Jr.
Skinner, H. J.
Slepian, Jos.
Stone, C. A.
Webster, E. S.
Wheeler, H. E.
Willien, Leon J., Jr.

Brockton—
Kendrick, T. E.

Brookline—
Howard, H.

Cambridge—
Dewey, Bradley
Forbes, Geo. S.
Goodwin, H. M.
Kalmus, H. T.
Knobel, Max
Lamb, A. B.
Little, A. D.
MacInnes, D. A.
Nickerson, W. E.
Richards, T. W.
Talbot, H. P.
Thompson, M. deK.
Walker, Wm. H.

Coolidge Corner—
Meyer, A. R.

Jamaica Plain—
Smith, F. T.

Lynn—
Devers, P. K.
Miller, L. B.
Nestor, J. F.

Malden—
Berry, E. R.

Marlboro—
McManus, Jos. D.

Newton Centre—
Pickard, G. W.
Walker, G. E.

North Dighton—
Griffin, M. L.

MASSACHUSETTS—Continued

Pittsfield—
Frank, J. J.
Kelly, J. F.
Moody, W. S.
Unger, M.
Woodside, F. C.

Quincy—
Rasmussen, F. J.

Salem—
Pitman, E. C.

Somerville—
Wilkins, H. S.

Southbridge—
Reeve, H. T.

So. Middleton—
Seifert, E. F.

Springfield—
Morris, A. W.

Swampscott—
Thomson, E.

Tufts College—
Durkee, F. W.

Waltham—
Flagg, F. P.

Watertown—
Coffin, C. F., Jr.

Waverley—
Horsch, W. G.

Williamstown—
Mears, Brainerd

Wollaston—
Ruppel, H. E. K.

Worcester—
Calhane, D. F.
Higgins, A. C.
Jeppson, G. N.
Marble, J. P.
Morse, H. E.
Saunders, L. E.

MICHIGAN

Ann Arbor—
Badger, W. L.
Baker, E. M.
Bartell, F. E.
Bigelow, S. L.
Ferguson, A. L.
Sturdevant, E. G.
Willard, H. H.

Bay City—
Hutchings, C. F.

Calumet—
Bosson, F. N.

Detroit—
Barnebey, O. L.
Bragg, C. T.
Cole, C. S.
Conner, A. B.
Crosby, E. L.
Flintermann, R. F.
Flumerfelt, O. F.
Frederick, W. A.
Ganschow, L. W.
Harig, Fred. C.
Hirshfeld, C. F.
Lane, H. M.
Lohr, J. M.
Marsh, A. L.

MICHIGAN—Continued

Detroit—Continued
Noyes, J. D.
Oswald, E. P.
Ramage, A. S.
Rhoads, A. E.
Schmelz, E. M.
Seifing, F. G.
Shepherd, F. A.
Skillman, Verne

Frankfort—
Evans, Ramiro

Houghton—
Carson, C. M.

Menominee—
White, A. R.

Midland—
Burdick, E. C.
Dow, H. H.
Gann, J. A.
Ward, L. E.

Sault Ste. Marie—
Harza, L. F.

Wyandotte—
Cranston, John
Hardcastle, Y. F.

MINNESOTA

Minneapolis—
Heisig, G. B.
Joselowitz, Goodwin
Mann, C. A.
Miller, L. F.

St. Paul—
Grelck, Wm.

MISSISSIPPI

Water Valley—
Wilcox, W. L.

MISSOURI

Columbia—
Marden, J. W.
Miller, F. H.
Schlundt, Herman

Fredericktown—
Mateer, T. J.

Joplin—
Rogers, C. B.

Kansas City—
Cherry, L. B.
Dalton, N. N.
Francis, P. B.
Kent, J. M.
Monroe, E. A.

Montgomery City—
Gill, J. P.

Mt. Washington—
Kryzanowsky, C. J.

St. Louis—
Graf, A. V.
Haskins, F. D.
Jackson, F. F.
Kohler, H. L.
Queeny, J. F.
Teeters, W. R.

Webb City—
Wallower, F. C.

MONTANA

Anaconda—
Frick, F. F.
O'Brien, A. L.

Baker—
Heinrich, E. G.

Billings—
Heath, R. F.

Butte—
Deshler, G. O.
Pulsifer, H. B.
Tower, R. E.

Great Falls—
Bardwell, E. S.
Burns, W. T.
Elton, J. O.
Maclay, E. G.

Rolla—
Sternberg, W. M.
Turner, W. D.

NEBRASKA

Omaha—
Hall, A. E.
O'Harra, B. M.
Low, W. H.
Randall, A. G.
Reinbold, Herman
Reinbold, Hugo

Valley—
Whitmore, J. D.

NEW HAMPSHIRE

Berlin—
Barton, C. B.
Comstock, R. L.
Fogarty, J. A.
McMurtrie, D. H.
Moore, H. K.
Richter, G. A.

Durham—
Perley, G. A.
Ross, E. S.

Wilton—
Abbott, Wm. G., Jr.

NEW JERSEY

Arlington—
Pickering, O. W.
Proctor, C. H.

Asbury Park—
Fernberger, H. M.
Wooley, W. R.

Bayonne—
Bjorkstedt, Wm.
Burgess, Louis
Corse, W. M.

Bloomfield—
Amer, H. S.
Braley, H. D.
Hageman, A. M.
Kleinfeldt, H. F.
McAllister, Paul
Romanelli, E.
Vassar, H. S.

Bound Brook—
Hemingway, F.
Saunders, H. F.

Butler—
Buttfield, A. C.

NEW JERSEY—Continued

Carteret—
Breckenridge, J. E.
Ford, F. E.

Chrome—
Clark, J. C.
Deacon, R. W.
Green, H. M.
Greenwood, H. D.
Hood, B. B.
Merrill, M. W.

Cranford—
White, J. H.

East Orange—
Bradley, Linn
Cowles, Harry D.
Driver, W. B.
Honan, E. M.
Koepping, E. D.
Mommo, E. J.
Prindle, E. J.
Prochazta, J. A.

Edgewater—
Grille, G. A., Jr.

Elizabeth—
Little, W. T.
Martens, Paul
Miller, A. B.
Pyne, F. R.
Ramsey, F. H.
Treacy, C. H.

Englewood—
Edwards, W. F.

Essex Falls—
Robb, Chas. D.

Gloucester City—
Knoedler, E. L.
Miner, H. S.

Haddon Heights—
Meyer, John

Harrison—
Hart, L. O.
Needham, H. H.

Haskell—
Cummings, Wm. J.

High Bridge—
Hall, John H.
Le Boutillier, Clement

Hoboken—
Bijur, Jos.
Hazeltine, L. A.
Winninghoff, W. J.

Irvington—
Bachofner, D. K.

Jersey City—
Baldwin, A. T.
Logan, M. H.
Lubowsky, Simon J.
Porth, H. W.
Swartley, H. R., Jr.

Kenilworth—
Lutz, G. A.

Keyport—
Schwarz, Edw.

Lawrenceville—
Willcox, Dudley

Leonia—
Folsom, R. A.

Maplewood—
Cunningham, F. W.
Wright, J. C.

NEW JERSEY—Continued

Merchantville—
Wickes, C. S.

Millstone—
Ruh, C. H.

Montclair—
Ellis, Carleton
Gesell, W. H.
Smith, Dyer

Morristown—
Anger, E. M.

Newark—
Barker, P.
Benjamin, E. O.
Boice, E. N.
Carter, F. E.
Ceader, Jos.
Colby, E. A.
Dewey, E. S.
Gifford, W. E.
Holding, H. H.
Hubley, W. F.
Liebschutz, M.
McNeill, Ralph
Peterson, F. H.
Robinson, F. W.
Sievering, Philip
Simon, C. K.
Walsh, P. C., Jr.
Weber, M. G.
Weston, E.
Youry, W. H. S.
Zwingenberger, O. K.

New Brunswick—
Bergen, R. C.
Dailey, J. G.
Keller, Oran
Philipp, Herbert
Richardson, L. T.
Voorhees, L. A.

North Bergen—
Van Keuren, W. L.

Orange—
Edison, T. A.
Kammerhoff, H. H.
Moore, C. W.
Sholes, C. E.

Parlin—
Holler, H. D.
Pitman, E. G.
Small, J. O.

Paterson—
Jarvis, E. G.

Pennsgrove—
Cooney, E. R.
Rhodin, B. E. F.

Perth Amboy—
Antisell, F. L.
Brown, M. J.
Fisher, H. W.
McNitt, R. J.
Peakes, G. L.
Roessler, F.
Rossi, Louis M.
Skowronski, S.
Stewart, M. E.
Temple, Sterling
Waring, T. D.

Phillipsburg—
Baker, John T.

NEW JERSEY—Continued

Plainfield—
Hall, H. M.
Hibbard, H. D.
Manahan, Paul R.
Pratt, H. A.
Spicer, C. W.

Princeton—
Hulett, G. A.
Northrup, E. F.
Smith, D. P.
Taylor, H. S.

Rahway—
Maeulen, Frederick
Miller, Daniel
Murray, B. L.

Roselle—
Grymes, E. S.

Rutherford—
Schroeder, C. M. E.

Sewaren—
Buttfield, W. J.
Cowles, A. H.

Short Hills—
Hough, Arthur

Silver Lake—
Cox, H. N.

South Orange—
Yunck, Carl I.
Yunck, J. A.

Summit—
Hornsey, J. W.

Trenton—
Brandt, M. F.
Harter, Wickham
Porter, H. F.
Redfield, C. S.
Saums, H. L.

Upper Montclair—
Buzby, A. D.

Watchung—
Moldenke, Richard

Weehawken—
Kraus, Ernest
Laise, C. A.
Schubert, B. H.

Westfield—
Alexander, H. H.
Carrier, C. F., Jr.

West Orange—
Pedersen, A. Z.
Worth, B. G., Jr.

Woodbridge—
Child, H. A.
Sargent, R. N.

NEW MEXICO

Carrizozo—
Wright, J. E.

Santa Fe—
Grubnau, G. M.

NEW YORK

Albany—
Adams, J. F.
Bainbridge, E. F.
Paulsson, Axel
Shiverick, M. D.
Case, T. W.
Junqueira, S.

NEW YORK—Continued

Batavia—
Walker, E. C., 3d

Bedford Hills—
Howe, H. M.

Beechurst (L. I.)
Landis, W. S.

Brooklyn—
Behnken, H. E.
Broms, H.
Carrier, S. C.
Cecere, T. J.
Cook, R. J.
Cowan, Wm. A.
Edwards, I. W.
Erhart, W. H.
Foster, O. R.
Frederick, G. E., Jr.
Hooker, S. C.
Kemper, D. A.
Langmuir, A. C.
Lodyguine, A.
Mahoney, J. N.
Pack, Chas.
Schultz, F. H.
Schultze, Paul
Scott, L. C.
Snyder, F. T.
Sperry, E. A.

Buffalo—
Albright, L.
Bierbaum, C. H.
Carrier, W. H.
Cummins, C. E.
Cushing, H. M.
Doty, E. L.
Drinker, Philip H.
Grotzinger, John
Harrington, R. N.
Kempke, C. W.
Knox, L. B.
Lawrence, J. N.
Mahlman, O. L.
Mattern, G. G.
Mitchell, R. W.
Mulligan, J. J.
Neal, J. R. H.
Patch, N. K. B.
Rippel, E. G.
Ruthenburg, Marcus
Schoellkopf, J. F., Jr.
Sicard, H. C.
Stephenson, H. L.
Strachan, E. K.
Theurer, G. A.
Ward, H. Lee
Wilke, Wm.
Wood, C. H.
Yager, J. J.
Zaremba, Edward

Canandaigua—
Phillips, Ross
Weeks, F. D.

Clinton—
Saunders, A. P.

Corning—
Sullivan, E. C.

Douglaston (L. I.)—
Vom Baur, C. H.

Flushing (L. I.)—
Bajda, J. J.
Watson, P. B.

NEW YORK—Continued

Fort Plain—
Fox, W. J.

Ilion—
Patterson, C. T.

Ithaca—
Barrett, W. R.
Gillett, H. W.

Kenwood—
Reeve, A. G.

LaSalle—
Gilbert, H. N.
Shields, J. E.
Wilson, N. A.

Lawrence (L. I.)—
Guiterman, K. S.

Little Falls—
Tinkler, L. G.

Lockport—
Howard, L. E.
Kenan, W. R., Jr.

Long Island City—
Reed, D. C.

Lyon Mountain—
Brakes, James

Massena—
Doerschuk, V. C.
Hewitt, A. C.
Mantel, Chas.
Whitaker, A. W., Jr.
Wilmore, C. B.

Monticella—
von Isakovics, Mary U

Mt. Vernon—
Dabolt, N. E.
Wilson, C. H.

New Brighton, S. I.
Blossom, E. L.
Walter, H. W.

New York City—
Abbé, P. O.
Acheson, E. G.
Addicks, L.
Aldridge, W. H.
Alley, J. C.
Alley, W. R.
Allyn, R. S.
Atwater, C. G.
Baker, H. A.
Barnes, H. H., Jr.
Barrows, F. E.
Barstow, W. S.
Baskerville, C.
Bateson, C. E. W.
Beck, E. A.
Bedell, E. H.
Bissell, R. W.
Boeck, P. A.
Bogue, C. J.
Bowman, W.
Brindley, Geo. F.
Brown, H. P.
Browne, deC. B.
Buck, H. W.
Buckie, R. H.
Bull, I. C.
Bumps, M. R.
Cameron, W. S.
Cardoza, J. B.
Carse, D. B.
Castle, S. N.
Chandler, C. F.
Clark, W. G.
Clark, W. J.

NEW YORK—Continued

New York City—Continued

Clevenger, G. H.
Cohen, F. W.
Coho, H. B.
Cohoe, W. P.
Colcord, F. F.
Cone, E. F.
Cooper, H. C.
Cooper, K. F.
Corning, C. R.
Crocker, J. R.
Czarnecki, F. C.
Daft, Leo
DeMiles, Paul
Deppe, W. P.
Dixon, Jos. L.
Doerflinger, W. F.
Doremus, C. A.
Dorr, J. V. N.
Dreyfus, W.
Drobegg, Gustave
Dutton, W. C.
Dwight, A. S.
Dyrssen, Waldemar
Eagle, H. Y.
Eckelmann, L. E.
Eimer, A.
Emerson, H.
Englehard, C.
Eurich, E. F.
Faber, H. B.
Featherstone, W. B.
Field, Crosby
Fink, Colin G.
Fitting, R. U.
Fitzgerald, W. V.
FitzGibbon, R.
Fleming, R.
Frank, K. G.
Freas, Thos. B.
Fries, H. H.
Gaines, R. H.
Glenn, E. R.
Goepel, C. P.
Gray, J. H.
Grosvenor, W. M.
Haas, S. W.
Halcomb, C. H.
Hale, E. W.
Hambuechen, Carl
Hampton, W. H.
Hansell, N. V.
Harris, J. W.
Hasslacher, J.
Hatzel, J. C.
Havill, O. A.
Hendrie, G. A.
Hendry, W. F.
Herty, C. H.
Hill, H. P.
Hill, N. S., Jr.
Hirsch, Alcan
Hirschland, F. H.
Hoge, J. F. D.
Hunt, A. M.
Huntoon, L. D.
Iliff, J. W.
Ingalls, W. R.
Iwata, Hiroshi
Jack, Geo. B., Jr.
Jacobson, B. H.
Jacoby, H. E.
Jicha, John
Jinguji, Genjiro
Kahn, Julius

NEW YORK—Continued

New York City—Continued

Keller, Edw.
Kellogg, A. O.
Keenan, T. J.
Kennedy, J. J.
Kern, E. F.
Keyes, D. B.
Kingsley, E. D.
Klein, O. H.
Klipstein, E. C.
Knapp, Geo. O.
Kohn, M. M.
Kunz, G. F.
Lake, D. B.
Leavitt, Wm. F. B.
Ledoux, A. R.
Lee, H. R.
Lee, S. T.
Liebmann, A. J.
Lloyd, R. M.
Loebell, H. O.
Lovejoy, D. R.
MacDonald, J. A.
Madsen, C. P.
Magnus, Benjamin
Maier, C. G.
Mailloux, C. O.
Manning, P. D.
Mantius, Otto
Marsh, C. W.
Martin, T. C.
Marvin, A. B.
Mason, F. S.
Mastick, S. C.
Mathewson, E. P.
Mays, S. W.
Maywald, F. J.
Mershon, R. D.
Metz, G. P.
Metz, H. A.
Miller, B. H.
Miller, D. D.
Moody, H. R.
Morehead, J. M.
Morgan, J. L. R.
Morrison, A. C.
Mortimer, J. D.
Mueser, E. E.
Muir, J. M.
Munn, J. C.
Murai, Ichiro
Muschenheim, F. A.
Myers, W. S.
Nichols, W. H.
Nichols, W. S.
Norton, W. S.
Onoda, N. P.
Parmelee, H. C.
Pasternak, Morris
Pennie, J. C.
Peters, F. F.
Petinot, N.
Poetschke, Paul
Pranke, Edw. J.
Price, E. F.
Prosser, H. A.
Raibourn, P. A.
Raimondo, Sebastiano
Randall, J. W. H.
Reed, S. A.
Reid, G. H.
Ricketts, L. D.
Riglander, M. M.
Riker, J. J.

NEW YORK—Continued

New York City—Continued

Riley, L. A., 2d
Roller, F. W.
Roth, C. F.
Rowand, L. G.
Ruffner, C. S.
Ruhl, L.
Ruhm, H. D.
Sachs, A. P.
Sargent, G. J.
Schloss, J. A.
Schuetz, F. F.
Schuyler, A. H.
Scott, E. K.
Seward, G. O.
Sharp, C. H.
Singer, C. E.
Smith, E. A. Cappelen
Smith, H. H.
Snook, H. C.
Southgate, G. T.
Sowers, Ossa
Spandow, W. E.
Speed, Buckner
Speiden, C. C.
Speirs, Chas. E.
Spicer, H. N.
Statham, Noel
Stevenson, Reston
Stockelbach, F. E.
Stone, J. S.
Stoughton, Bradley
Strauss, F. A.
Swenarton, W. H.
Takamine, J.
Takamine, J., Jr.
Takikawa, Y.
Tate, A. O.
Teeple, J. E.
Thatcher, C. J.
Thompson, J.
Toch, M.
Treadwell, J. C.
Tucker, S. A.
Van Arsdale, G. D.
Vogeler, W. R.
Waddell, M.
Wadleigh, F. R.
Wahlberg, Axel F.
Waldo, W. B.
Walker, A. L.
Walker, R. G.
Warner, F. H.
Washburn, F. S.
Washburn, W. F.
Watanabe, H.
Waterman, F. N.
Weintraub, Ezechiel
Westervelt, W. Y.
Wheeler, A. E.
Whitaker, M. C.
White, J. G.
Wigglesworth, H.
Wilkinson, W. N.
Williams, A.
Williams, L. W.
Winship, W. W.
Witherell, C. S.
Wolf, H. J.
Wood, E. F.
Wright, Arthur
Zeller, R.
Zons, F. W.

NEW YORK—Continued

Niagara Falls—

Acheson, E. G., Jr.
Allen, D. E.
Avery, J. M.
Barton, P. P.
Beatty, H. H.
Becket, F. M.
Bliss, Wm. Lord
Brallier, P. S.
Brown, R. G.
Campbell, Geo.
Carveth, H. R.
Chace, R. T.
Cole, E. R.
Converse, V. G.
Cox, G. E.
Critchett, J. H.
Dunlap, O. E.
Edmands, I. R.
FitzGerald, F. A. J.
Fowler, R. E.
Fuller, G. P.
Gegenheimer, R. E.
Glaze, John B.
Glennie, R. D.
Griffith, J. R.
Guinther, John
Hamann, A. M.
Hardie, C. G.
Harper, J. L.
Hartmann, M. L.
Hauman, E. L.
Herzog, G. K.
Hills, L. H.
Hinckley, A. T.
Hinman, R. G.
Hooker, A. H.
Hutchins, Otis
Imlay, L. E.
Johnson, J. A.
Judson, L. C.
Kellogg, H. W.
Kemmer, F. R.
King, J. A.
Koethen, F. L.
Kokatnur, V. R.
Lansing, C. N.
Lavene, H. A.
Lidbury, F. A.
Low, F. S.
Lyster, T. L. B.
MacMahon, J. D.
MacMillan, J. R.
Marshall, J. G.
Mauran, M.
McKnight, W. A.
McMillen, Herbert
Meredith, W. F.
Miller, D. R.
Moritz, C. H.
Morley, M. H.
Moyer, G. C.
Muggleton, G. D.
Noyes, H. L.
Nutting, E. G.
Oatman, A. B.
Ohliger, L. P.
Osborne, S. E.
Patterson, L. G.
Patterson, T. A.
Plock, A. F.
Ralston, O. C.
Rossi, A. J.
Rowland, J. M.

NEW YORK—Continued

Niagara Falls—Continued
Rykenboer, E. A.
Savage, P. G.
Schoellkopf, P. A.
Sergeant, E. M.
Sims, C. E.
Simmers, A. L.
Simonsen, I. B.
Smith, Acheson
Smith, E. S.
Snowdon, R. C.
Speiden, E. C.
Stamps, F. A.
Stillesen, J. M. A.
Stone, G. W.
Sullivan, A. P.
Tone, F. J.
Uptegraff, T. M.
Vaughn, C. F.
Wallace, W.
Wescott, E. W.
White, R. H.
Williams, C. E.
Williamson, A. M.
Wilson, I. W.
Wilson, W. K.
Yngve, Victor

Nyack—
Betts, A. G.

Oneida—
Bailey, R. O.

Ossining—
Acker, Chas. E.

Pelham—
Brown, C. J.

Penn Yan—
Taylor, C. E.

Plattsburg—
Sheffield, W. T.

Potsdam—
Russell, L. K.

Prince Bay (L. I.)—
Johnston, F. A.

Richmond Hill (L. I.)—
Haslwanter, C.
Herreshoff, J. B., Jr.
Voss, Wm.

Rochester—
Brown, C. G.
Burch, W. S.
Miller, S. W.
Schwarz, R. C.
Will, R. T.

Rome—
Parish, R. R.

Schenectady—
Andrews, Mary R.
Andrews, W. S.
Arsem, W. C.
Berg, E. J.
Coffin, F. P.
Collins, E. F.
Coolidge, Wm. D.
Creighton, E. E. F.
Dantsizen, Christian
Dushman, Saul
Hawkins, L. A.
Kienle, R. H.
Langmuir, I.

NEW YORK—Continued

Schenectady—Continued
Lof, E. A.
McTerney, P. A.
Murphy, E. J.
Ohashi, F.
Reist, H. G.
Rice, E. W., Jr.
Rushmore, D. B.
Seede, J. A.
Steinmetz, C. P.
Taylor, J. B.
Thurston, L. S.
Van Brunt, C.
Whitney, W. R.
Willey, L. M.
Winder, C. A.
Winne, H. A.

Stony Point—
Hood, H. P.

Syracuse—
Berry, G. M.
Brookfield, W. B.
Conklin, E. B.
Handy, B. H.
Harvey, F. A.
Houghton, A. C.
Mathews, J. A.
Newkirk, E. D.
Pearce, W. C.
Pennock, J. D.

Tompkinson (L. I.)—
Berger, Alfred

Tottenville (S. I.)—
Wagner, C. S.

Troy—
Bryson, T. A.
Hunter, M. A.
Lincoln, A. T.
Patton, D. C.

Utica—
Steele, F. B.

Watertown—
Wilder, M. S.

Yonkers—
Baekeland, L. H.
Duurloo, F.
Eldridge, C. H.
Fish, Job, Jr.
Morey, S. R.

NORTH CAROLINA

Badin—
Parks, R. E.

Charlotte—
Gilchrist, P. S.
Lee, W. S.

Clemmons—
Douthit, J. B.
Pickens, Rufus H.

OHIO

Akron—
Dinsmore, R. P.
Gould, S. P.
Knight, M. A.
Shaw, E. C.

Alliance—
Baily, T. F.
Cope, F. T.
Foster, C. L.

OHIO—Continued

Barberton—
Austin, A. O.
Lyons, H. N.

Canton—
Buckwalter, T. V.
MacGregor, Walter

Cincinnati—
Bray, C. A.
Davison, A. W.
Dittmar, Carl
Ecker, Howard, Jr.
Elliott, Geo. K.
Pugh, A. H., Jr.

Cleveland—
Barron, A. N.
Blake, H. D.
Burwell, A. W.
Chaffe, J. B., Jr.
Clymer, W. R.
Fahrenwald, F. A.
Feild, A. L.
Goss, B. C.
Graves, W. G.
Hamister, V. C. H.
Herron, J. H.
Holmes, M. E.
Hyde, E. P.
Keen, Wm. H.
Koehler, W.
Kranz, W. G.
Marshall, G. G.
Martin, J. W., Jr.
Megroot, J. P.
Merrill, G. S.
Pratt, E. B.
Richardson, E. A.
Rittenhouse, Edw.
Schmidt, J. H.
Smith, A. W.
Tower, O. F.
Wallau, H. L.
Wellman, S. T.
Whitlock, E. H.
Woodward, J. M.

Columbus—
Demorest, D. J.
Lower, J. R.
Schaaf, Downs
Withrow, J. R.

Coshocton—
Howell, C. H.

Dayton—
Clements, F. O.
Fox, C. P.
Hummert, R. H.
May, R. B.
Niswonger, E. E.
Suman, R. G.

Fostoria—
Downes, A. C.

Fremont—
Brooks, H. W.
Goodwin, J. H.

Galion—
McBerty, F. R.

Lakewood—
Brooks, W. C.
Bullock, A. R.
Chaney, N. K.
Drefahl, L. C.

OHIO—Continued

Lakewood—Continued
Gillingham, C. A.
Hazelett, C. W.
Kuhn, H. M.
Mott, W. R.
Ordway, D. L.
Pulman, O. S.
Reid, R. R.
Russell, C. A.
Waitt, W. G.

Lancaster—
Frink, R. L.

Mansfield—
McBerty, F. H.
Pence, M. F.
Wolf, F. L.

Middletown—
Ahlbrandt, G. F.
Beck, W. J.
Eldridge, S. E.

Napoleon—
Long, G. E.

Norwood—
Bell, Wm. H.

Painesville—
Wells, H. P.

Portsmouth—
Kinnear, H. B.
Wooldridge, W. J.

Ravenna—
Mann, W. W.

Rittman—
Laib, Walter

Salem—
Davis, D. L.
Eberwein, S. J.
McKeown, Wm.

Steubenville—
Glemmitt, W. B.

Toledo—
Moorhouse, L. B.
Nagel, W. G.
West, P. A.

Wadsworth—
Shapiro, H.

Youngstown—
Russell, D. A.

OKLAHOMA

Bartlesville—
Born, Sidney

Shawnee—
Dodge, W. E.

Tulsa—
Higgins, E. C., Jr.
Kroll, Cornelius
Miller, Walter
Taber, G. H., Jr.

OREGON

Corvallis—
Strong, R. K.

Portland—
Hall, E. L.
Morrison, W. L.

PENNSYLVANIA

Allentown—
Snelling, W. O.

Altoona—
Casselberry, H.

Ambridge—
Farnham, F. F.

Aspinwall—
Hedden, S. E.
Hessom, B. F., Jr.
McMillen, R. H.
O'Donovan, J. S.

Bellefonte—
Ward, A. T.

Bethlehem—
Buck, C. A.
Burgess, C. W.
Butts, Allison
Fehnel, J. Wm.
Hommel, R. P.
Kay, M. J.
Lehr, H. D.
Luckenbach, L. J.
MacNutt, Barry
Richards, J. W.
Roush, G. A.
Seyfert, S. S.
Smith, F. S.
Smull, J. G.
Spurrier, J. R.
Uihlein, R. A.

Brackenridge—
Connell, H. R.
Ober, J. E.

Bridgeville—
Anderson, A. N.
Grossmann, M. A.
Saklatwalla, B. D.

Canonsburg—
Nicholson, K. C.
Vogt, L. F.

Chester—
Comey, A. M.
Krause, W. B.

Conshohocken—
Davis, F. W.

Coraopolis—
Hensen, Emil
Stafford, S. G.

Creighton—
Cox, S. F.
Gelstharp, F.

Cynwyd—
Parkhurst, C. W.

Duquesne—
Burroughs, D. C.
Cummins, A. C.
Eakin, C. T.
Stewart, H. M.

Easton—
Adamson, G. P.
Buhl, Wm.
Chapin, H. C.
Gordon, C. McC.
Hart, E.
Shimer, E. B.

Edgewood Park—
Scott, W. S.

Ellwood City—
Dunn, J. J.
Offutt, J. W.

PENNSYLVANIA—Continued

Erie—
Valentine, I. R.

Gettysburg—
Parsons, L. A.

Greensburg—
Koppitz, C. G.

Grove City—
Welker, J. A.

Hamburg—
Reese, P. P.

Harrisburg—
Kinter, G. R.
Orr, C. A.
Smith, J. H.

Hellertown—
Barrows, W. A.

Jenkintown—
Davis, R. W., Jr.

Latrobe—
Garratt, Frank
MacFarland, A. F.
Sherk, H. C.

Lebanon—
Weimer, E. A.

Mars—
Adair, W. R.

McKeesport—
Goodspeed, G. M.

Merion Station—
Boericke, Harold

Monessen—
Owens, E. W.

Mount Airy—
Hall, C. A.

Mount Penn—
Kramer, L. B.

Natrona—
Darlington, H. T.
Huff, E. L.

New Kensington—
Bakken, H. E.
Bell, A. D.
Blough, Earl
Churchill, H. V.
Hamor, W. A.
Milligan, L. H.
Moormann, T. A.

Oakmont—
Frary, F. C.
Hitner, H. F.
Moore, J. R.

Oil City—
Bayless, F. B.

Palmerton—
Breyer, F. G.
Holstein, L. S.
Vanderwaart, P. T.

Parkesburg—
Beale, H. A.

Philadelphia—
Asef, Waldemar
Bash, F. E.
Bassett, H. P.
Benoliel, S. D.
Blasius, C. E.
Bonine, Chas. E.
Bright, A. C.
Brophy, O.

PENNSYLVANIA—Continued

Philadelphia—Continued

Brown, R. P.
Bustos, Enrique
Callen, A. S.
Canfield, J. M., Jr.
Chillas, R. B.
Clamer, G. H.
Corbin, J. Ross
Dalton, Walter
Devereux, W.
Eglin, W. C. L.
Fletcher, W. E.
Flinn, E. H.
Frickey, R. E.
Fulweiler, W. H.
Furness, R.
Gailey, A. J.
Gellert, N. H.
Gibbs, A. E.
Gould, D. F.
Haig, J. E.
Harrison, W. E.
Hering, C.
Hess, H.
Hitchcock, F. R. M.
Hodson, Frank
Holland, W. E.
Hollander, C. S.
Howard, G. M.
James, W. F.
Johnson, W. McA.
Keith, N. S.
Kent, S. L.
Kummer, M. T.
Kutz, Milton
Lafore, J. A.
Laird, K. V.
Lavino, E. J.
Levin, I. H.
Lucas, C. C., Jr.
Lukens, H. S.
Mahlman, O. L.
Mainwaring, W. D.
McConnell, J. Y.
Meigs, C. C.
Merzbacher, Aaron
Moerk, F. N.
Mottinger, B. T.
Neville, Neil
Oldach, F. W.
Paul, H. N.
Peirson, C. L.
Reich, J. S.
Rosenfeld, J. R.
Rosengarten, G. D.
Russell, C. J.
Ryan, F. J.
Sadtler, S. P.
Sadtler, S. S.
Salom, P. G.
Schamberg, M.
Scott, W. M.
Smith, E. F.
Smith, E. W.
Smith, I. B.
Stevens, J. F.
Styri, Haakon
Taggart, W. T.
Taylor, H. N.
Thompson, C. M., Jr
Thwing, C. B.
Weeks, C. A.
Weisenburg, Andrew
Williams, H. J.
Wirt, Chas.

PENNSYLVANIA—Continued

Pittsburgh—

Aston, James
Bacon, R. F.
Blakeslee, D. W.
Brown, John T., Jr.
Brownlee, R. H.
Clarke, E. B.
Crabtree, F.
Crawford, C. A.
Darrin, Marc
Demmler, A. W.
Dougherty, J. W.
Dunn, H. E.
Edgerton, C. T.
Eschholz, O. H.
Flannery, Jas. J.
Gibson, C. B.
Goodale, S. L.
Graybill, J. H.
Graef, W. H.
Grumbling, J. S.
Gruse, W. A.
Hartley, R. H.
Hecker, Chas.
Higgins, D. F.
Hill, C. W.
Hitchcock, H. K.
James, J. H.
Jones, G. W.
Kemery, P.
Kier, S. M.
Laughlin, H. H.
Lincoln, P. M.
Lindsay, W. H.
Loeffler, Geo. O.
MacRae, Duncan
Mathias, D. L.
McDonald, R. A.
McKinley, Jos.
McNiff, G. P.
Meyers, H. H.
Moore, R. W. E.
Moore, W. E.
O'Neil, R. D.
Page, G. S.
Pinkerton, A.
Pope, Chas. E.
Robbins, Chas.
Rottmann, C. J.
Schluederberg, C. G.
Schoepf, T. H.
Silverman, A.
Skinner, C. E.
Slocum, F. L.
Snyder, C. G.
Speller, F. N.
Stone, E. C.
Stupakoff, S. H.
Tafel, Theodore, Jr.
Taylerson, E. S.
Turnock, L. C.
Uhlenhaut, F., Jr.
Unger, J. S.
Viol, C. H.
Weidlein, E. R.
Wiley, B.
Yardley, J. L. McK.
Zimmerman, R. E.

Primos—

Boericke, G.

Pulaski—

McCullough, H. F.

Reading—

Shindell, H. F.

PENNSYLVANIA—Continued

Roaring Springs—
McDonald, F.

Scranton—
Winkel, Herbert

State College—
Armstrong, H. M.
Chedsey, W. R.
Dudley, Boyd, Jr.
Soley, W. G.

Steelton—
Reed, J. C.

St. Marys—
Fleming, S. H.
Smith, G. S.
Wilson, G. C.

Swarthmore—
Alleman, G.
Creighton, H. J.

Swissvale—
Lewis, J. D.

Tamaqua—
Brown, K. R.
Burt, M. C.
McQuaid, H. S.

Tarentum—
Parkinson, J. C.

Titusville—
Evans, C. T.

Tyrone—
Woodbridge, W. B.

Verona—
Rodman, Hugh

Washington—
Arnold, H. C.
Johnson, Jesse
Sargent, G. W.
Weitzenkorn, J. W.

Wilkinsburg—
Lewis, J. W.
O'Neill, W. J.
Wills, W. H., Jr.

Wyomissing—
Parker, J. H.

York—
Smith, Lyon

RHODE ISLAND

Providence—
Andrews, J. H.
Nagelvoort, Adriaan
Saunders, W. M.
Thurber, J. H.
Wilson, Ellery S.

SOUTH CAROLINA

Charleston—
Hughes, H.

Columbia—
Mills, J. E.

Sumter—
Van Deventer, H. R.

SOUTH DAKOTA

Deadwood—
Boyer, Lee

TENNESSEE

Brentwood—
Hogan, F. W.

TENNESSEE—Continued

Bristol—
Hiddleson, W. A.

Chattanooga—
Davison, G. L.
Kruesi, P. J.
Taylor, W. W.
Waldo, W. G.
Wiggins, C. Q.

Clarksville—
Foust, T. B.

Knoxville—
Sammons, W. R.

Maryville—
Adams, T. J.
Mitman, W. T.
Swany, G. R.

New Market—
Livingston, W. J., Jr.

TEXAS

Austin—
Harper, H. W.
Schoch, E. P.
Walker, T. B.

Beaumont—
Hutchison, J. B.

El Paso—
Fraser, Lee
Schimerka, F. S.
Stevens, B.

Fort Worth—
Eastman, H. M.

Houston—
Alexander, C. M.
Henst, J. Vander
Weiser, H. B.

Palestine—
Hunt, N. H.

Port Arthur—
Roberts, G. I.
Slater, W. A.
Stockton-Abbott, L.

San Antonio—
Shaw, S. F.
Williamson, P. B.

Somerville—
Kroemer, F. W.

Wichita Falls—
Bowman, F. C.
Haynes, J. H.

UTAH

La Sal—
Morgan, H. J.

Midvale—
Cullen, J. F.
Hamilton, E. H.

Pleasant Grove—
Hayes, J. J.

Provo City—
Smith, F. W.

Salt Lake City—
Cameron, F. K.
Fleming, E. P.
Hansen, C. A.
Putnam, W. R.
Wright, C. A.

VERMONT

Northfield—
Howard, S. F.

VIRGINIA

Alexandria—
Fawcett, L. H.

Hampton Roads—
Meineke, O. H.

Low Moor—
Davison, A. W.

Norfolk—
Webb, L. W.

Richmond—
Walters, Jos.

Roanoke—
Brown, F. C., Jr.

Saltville—
MacMahon, J. H.

University—
Dunnington, F. P.

WASHINGTON

Chewelah—
Dolman, C. D.

Hoquiam—
Lamb, F. H.

McCleary—
Macke, J. C.

Seattle—
Corey, C. R.
Gailey, W. R.
Magnussen, C. E.
Miller, A. A.
Peterson, J. C.
Thomas, B.

Spokane—
Armstrong, L. K.
Keffer, Frederick
King, Rowland
Levy, G. J.

Tacoma—
Dunlap, T. E.
Everette, W. E.
MacPherson, A. R.
Porro, T. J.
Zischkau, O. C.

WEST VIRGINIA

Blue Field—
Finks, G. H.

WEST VIRGINIA—Continued

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App, J. C.
Palmer, C. S.
Pierce, J. B., Jr.
Pitcher, A. M.
Rollin, Hugh

Fairmont—
McQuillin, Francis

Martinsburg—
Kohlhausen, B. F.

Morgantown—
Clark, F. E.
Hite, B. H.

Warwood—
Bumgardner, J. W.

WISCONSIN

Carrollville—
Ceresole, M. A.

Eau Claire—
Thompson, J. G.

Jamesville—
Stewart, C. O.

Kenosha—
Evans, G. D.

Madison—
Burgess, C. F.
Helfrecht, A. J.
Kahlenberg, L.
Kowalke, O. L.
Ruhoff, O. E.
Schulte, W. B.
Storey, O. W.
Tan, Bo N.
Watts, O. P.

Milwaukee—
Adams, J. L., Jr.
Bovee, B. A.
Chadwick, R. A., Jr.
Evans, C. T.
Kremers, J. G.
Nash, C. A.
Raeth, F. C.
Wesle, E. H.
Zimmermann, J. G.

Oconomowoc—
Ryan, R. W.

WYOMING

Casper—
McKim, J. W.

Thermopolis—
Freeman, G. N.

CANADA

BRITISH COLUMBIA

Anyox—
Speight, H.

Nelson—
Fowler, S. S.

Trail—
Beasley, F. E.
Lee, F. E.
Lytle, L. B.
Willis, F. S.

Vancouver—
Barwick, W. S.
de Giovellina, E. C.
Haggen, E. A.
Hayward, R. F.
McIntosh, D.

MANITOBA

Winnipeg—
Armes, H. P.
Brown, G. J.

NEW BRUNSWICK

St. John—
Vaughan, F. P.

NEWFOUNDLAND

Grand Falls—
Judge, T. F.

NOVA SCOTIA

New Glasgow—
Cantley, T.

CANADA—Continued

ONTARIO

Dryden—
Mathews, F. P.
Goderich—
Kelleher, James
Kakabeka Falls—
Farrow, P. R.
Kingston—
Cadenhead, A. F. G.
Goodwin, L. F.
Goodwin, W. L.
Merrickville—
McMullen, G. P.
Merritton—
Hedalen, John
Niagara Falls—
Arson, W. H.
Doyle, H. L.
Gardner, G. N.
Nickelton—
Gillis, J. H.
Ojibway—
Baltzell, W. H.
Orangeville—
Deagle, L. M.
Orillia—
Lamble, B. C.
Tudhope, H. R.
Ottawa—
Frost, G. B.
Renfrew—
Cram, W. C., Jr.
Sandwich—
Walker, G. H. P.
St. Catharines—
Sieger, G. N.
Sudbury—
Morin, H. A.
Thorold—
Schabacker, H. E.
Toronto—
Allan, W. G.
Bain, J. W.
Baldwin, J. C. B.
Barrows, W. S.
Benson, G. C.
Brown, N. B.
Burpee, L.
Burt-Gerrans, J. T.
Clark, F. C.
Crafts, W. N.
Gaby, F. A.
Gifford, W. M.
Guess, G. A.
Hedstrom, E. S.

ONTARIO—Continued

Toronto—Continued
Kenrick, F. B.
MacDougall, A. J.
Marshall, A. L.
Miller, W. L.
Moffat, J. W.
Robertson, F. D. S.
Roumbanis, D. C.
Westman, L. E.
Wood, E. M.
Welland—
Eaton, I. C.
Renton, W. L.
Turnbull, R.
Young, John
Windsor—
Henderson, E. G.
Vorce, L. D.

QUEBEC

Buckingham
Hambley, F. J.
Chandler—
Hedin, Jos. E.
Deschenes—
Strom, Birger
Westby, G. C.
Hull—
Bennie, P. McN.
Wilson, J. R.
Longuevil—
Platts, J. C.
Montreal—
Davidson, T. R.
Eardley-Wilmot, T.
Falter, P. H.
Gilman, M. C.
Janssen, W. A.
Kaelin, F. T.
Matheson, H. W.
Medbury, C. F.
Morgan, N. L.
Stansfield, A.
Wales, J. A.
Woodyatt, J. B.
Ottawa—
Haanel, E.
Simpson, Louis
Shawinigan Falls—
Acton, E. H.
Crowther, C. W. W.
Witherspoon, R. A.
Westmount—
Spencer, A. G.

CUBA

Guantanamo—
Simon, E. J.
Havana—
De Milan, Alex
Font, G. Y.

Havana—Continued
Smith, J. M.

Miranda—
Hill, R. H.

MEXICO

Mexico City—
DeLandero, C. F.
Hinton, G. B.
Rost, H. F.

Monterrey—
Berthier, U. H.
Orizaba—
Wilkins, E. M.

PANAMA

Gatun—Grier, B. R.

SOUTH AMERICA

BOLIVIA

Oruro—
Ferron, R. D.

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De Medeiros, T. S. V.
S. Paulo—
Bowles, R. H.
Byington, A. J.
De Souza, E.
Pompeia, Jonas
Villa Nova de Lima—
Jones, H.
Wilder, F. L.

CHILE

Antofagasta—
Rowe, G. M. W.
Chuquicamata—
Jörgensen, E. L.
McClenahan, J. S.
Oldright, G. L.

CHILE—Continued

Iquique—
Boesch, J. E.

Santiago—
Cardoen, Remy
Carr, H. D.
Diaz-Ossa, B.

Valparaiso—
Hobsbawn, I. B.

PERU

Arequipa—
Rosenshine, L. J.

Morococha—
Dalmeida, J. A.

Oroya—
Mather, T. W.

AUSTRALIA

NEW SOUTH WALES

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Du Faur, J. B.
Port Kembla—
White, E. A.
Sydney—
Murphy, R. K.
Wallaroo—
Hancock, H. L.

NEW ZEALAND

Auckland—
Crookes, S. T.
Christ Church—
Philpott, H. E. R.
Stark, E. E.
Dunedin—
Shepherd, F. R.

QUEENSLAND

Brisbane—
Henderson, J. B.
Jackson, A. G.
Mt. Morgan—
Johns, M. J.

TASMANIA

Hobart—
Butters, J. G.
Gepp, H. W.
Queenstown—
Lawrenson, J. S.
Sticht, R. C.

VICTORIA

Melbourne—
Chappell, W. C.
Gillies, P. M.

ASIA

CHINA

Canton—
Laird, C. N.
Hankow—
Wang, C. Y.
Hanyang—
Woo, Z. T. K.
Hong Kong—
Lee, L. P.
Wong, K. T.
Lincheng—
Kwang, K. Y.
Shanghai—
Gaither, R. H.
Czechuen—
Chiang, Y. K.
Tayeh—
Wong, W. A.

INDIA

Bangalore—
Srinivasayengar, V. K.
Calcutta—
Eldredge, M.

JAPAN

Akita—
Ikeda, K.
Satoh, H.
Echigo—
Nakamura, Yushichiro
Ehimeken—
Yabe, C.
Fuknoka—
Kaneko, K.
Tatsumi, E.
Fukushimaken—
Nakashima, Shigemaro
Hyogo-ken—
Murahashi, S.
Ishikawaken—
Iwai, Kyosuke
Komatsu—
Yano, M.
Kanagawa-ken—
Tabata, Kozo
Kawasaki—
Matsushita, N.
Shinjo, Y.

ASIA—Continued.

JAPAN—Continued

Kobe—
Isobe, F.
Tachihara, Jin
Tada, S.

Kumamoto—
Fukuda, M.

Kyoto—
Nakasawa, Yoshio
Namba, M.
Nishikawa, Kikei
Yoshikawa, K.

Nagano—
Takahashi, Taketaro

Nagoya—
Hori, S.
Mine, S.

Nikko—
Kaku, Juroku

Osaka—
Arakawa, E.
Itoh, Ichiro
Kimura, Z.
Kobayashi, W.
Tsujimoto, K.
Yunoki, S.

Sendai—
Fuseya, G.

JAPAN—Continued

Shigaken—
Kurahashi, T.

Tokohama—
Tada, K.

Tokyo—
Gotoh, Issaku
Higashi, Sentaro
Igarashi, T.
Inui, K.
Ishikawa, Ichiro
Ito, Ichiro
Kai, Hideo
Kameyama, Naoto
Kato, Y.
Katsura, B.
Kawamura, T.
Kawakita, Y.
Kitawaki, I.
Kishi, K.
Nakahara, S.
Nakao, K.
Nishida, Hirotaro
Nohara, T.
Oshima, Y.
Shigematsu, Y.
Takakuwa, K.
Yamasaki, J.
Yamaguchi, K.
Tada, Y.

AFRICA.

CAPE COLONY

Somerset, West—Quinan, K. B.

EUROPE.

BELGIUM

Antwerp—
Deakin, Gerald

Brussels—
Cito, C. C.
Cousin, Jules
Solvay, A.

Jemeppe—
Queneau, A. L. J.

Mons—
Henault, O-Dony.

Neerpelt—
Raeder, Bjorn

FINLAND

Abo—
Aminoff, G.

FRANCE

Paris—
Allen, O. F.
Altmayer, M. E.
Caudrelier, E.
deGeofroy, A.
DeSallier, Rene
Gall, H.
Halter, Georges
Keller, C. A.
Marie, C.
Ostheimer, J. W.
Roberts, W. S.

Seine-et-Oise—
Humbert, E. P.

GREAT BRITAIN

Birmingham—
Threlfall, R.

Bristol—
Brander, J.

Burton-on-Trent—
Winkley, S. F.

Codsall—
Castle, G. C.

Essex—
Jones, H. A.

Glasgow—
Lackie, W. W.
Scott-Maxwell, J. M.

Hatch End—
Jacob, Arthur

Irvine (Scotland)—
Walker, J. W.

Liverpool—
Smith, A. T.

London—
Ashcroft, E. A.
Berk, P. F.
Brown, P. Hunter
Carnegie, E.
Chalas, A.
Cornthwaite, Haydn
Goldstein, P.
Hadfield, R. A.
Johnson, Arthur
MacKness, C. F.
Merz, C. H.
Oakden, W. E.
Sprague, E. C.
Shields, John
Walters, George

EUROPE—Continued.

GREAT BRITAIN—Continued

Manchester—
Pring, J. N.

Northwich—
Johnson, A.

Sheffield—
Cutts, V. O.
Etchells, H. E.
Fawcett, P.
Klouman, H.
Rowland, T.

Spondon—
Williams, E. R.

Sunbury-on-Thames—
Cowper-Coles

Tunbridge Wells—
Colquhoun, James

Walthamstow—
Wilder, F. L.

Widner—
Pritchard, D. A.

ITALY

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Merizzi, G.
Paravicini, R.
Rossi, C.
Toniolo, Carlo

Rome—
Catani, Remo
Chiaraviglio, D.
Morani, Fausto

Turin—
Gardiol, A. D.
Giolitti, F.

NORWAY

Bergen—
Anderson, F.
Turner, M. R.
Tysland, Geo.

Borgestad—
Knudsen, Rolf

Christiania—
Berg-Hansen
Bjornson, E.
Bryn, K.
Collett, E.
Collett, Ove
Dedichen, Herman
Farup, P.
Goldschmidt, Heinrich
Gronningsater, Anton
Hiorth, A.
Hiorth, F. V. L.
Hole, Ivar
Homan, C. H.
Hurum, F.
Johansen, G. H.
Krog, K. M.
Olsen, T. S.
Saeter, H. B.
Somdal, J. A.
Stephanus, A. D.
Tharaldsen, Filip
Thorne, C. A.
Von Krogh, Johan

NORWAY—Continued

Christiansand—
Frisak, Arvid
Lenschow, H. H.

Drammen—
Sturbelle, L. C.

Fredriksstad—
Moltkehansen, I. J.

Hoyanger—
Murer, Johan

Nakkerud—
Loken, R.

Odda—
Andersen, Jorgen
Giersten, S.

Rjukan—
Bonnevie, H.
Nilssen, Bjarne

Ryfylke—
Sneath, W. H.

SPAIN

Barcelona—
Lana, C.

Reys—
Miro, A. R.

Tarrasa—
Balta de Cela, Jose

SWEDEN

Djursholm—
Berglund, E. S.

Falun—
Yngstrom, L.

Gullspang—
Hedlund, M.

Sodertelje—
Tesch, H. G. A.

Stockholm—
Carlson, Birger
Harden, J.
Kring, Oskar
Leffler, J. A.
Lilja, S. G.
Lindberg, S. C.
Ljungh, Hjalmar
Martin, Herbert
Olsson, H.
Palen, A. G. P.
Palmaer, K. W.

Trollhattan—
Forssell, J.

Vesteras—
Edstrom, J. S.
Torell, T. F.

Westervik—
Tillberg, E. W.

SWITZERLAND

Basel—
Engi, Gadient
Fichter-Bernoulli, F.

Geneva—
Gandillon, A.
Guye, P. A.
Lacroix, H.

Turgi—
Landolt, H.

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THE ACTIVATION OF CARBON[1]

By N. K. Chaney.[2]

The war has brought into existence, as an article of commerce, a new product known as "activated carbon."

Originated as a means of defense against toxic gases in warfare this material is rapidly finding industrial applications, the extent and novelty of which are not as yet generally appreciated.

The development of activated carbon represents something more than a mere improvement in older forms of adsorptive carbon, or in older processes. It stands for a new viewpoint as to the general nature of so-called amorphous carbon.

Carbon has been hitherto defined as existing in three distinct modifications, diamond, graphite and amorphous. The latter term has comprised a heterogeneous assortment of natural and artificial carbon materials, possessing a variety of widely different physical and electrical properties, such as manifestly could not be explained by a single allotropic modification of elementary carbon.

Thus, while the complexity of so-called amorphous carbon has long been recognized, we are beginning for the first time to obtain a rational idea of its individual components and their relations to one another.

Some of these ideas and relationships have been indicated in a general way in a recent joint paper[3] by A. B. Lamb, R. E. Wilson and the writer.

In other accounts of the work of the Bureau of Mines[4] and of the Chemical Warfare Service[5] are related the circumstances which led the National Carbon Co. to devote its Research Laboratory to the development of a satisfactory adsorbent charcoal for the American Army.

[1] Manuscript received August 14, 1919.

[2] Assistant Director, Research and Development Laboratories, National Carbon Co., Inc., Cleveland, Ohio, and Consulting Chemist, Research Division, C. W. S., in charge Charcoal Research Unit.

[3] J. Ind. and Eng. Chem. (1919), **11**, 420-467, July number.

[4] Bull. 178-A War Gas Investigations. Bureau of Mines.

[5] J. Ind. and Eng. Chem. (1919), **11**, Jan. and April numbers.

In accepting this problem, contrary to the popular impression, we possessed no special knowledge of vegetable charcoals, or special acquaintance with the processes and literature regarding the preparation of adsorbent and clarifying materials.

We assumed at the start that whatever general knowledge existed in these fields was inadequate to a solution of the problem otherwise the stress of two years' gas warfare would have brought about solutions of which the Government would have had adequate information. We were strangely ill-informed of the partial success already obtained by England, France and Germany.

Thus the belief that no adequate protection existed against certain toxic gases, and our lack of information regarding the methods and results obtained by others in this field provided the compelling stimulus for a new and fundamental investigation of the first principles of adsorptive activity in carbon.

The result of this intensive research was the gradual development of a broad general conception as to the nature of active carbon and its relations to the generally occurring forms of carbon.

This general theory in its complete form rests upon two independent, experimentally established postulates.

(1) That elementary carbon (other than diamond and graphite) exists in two modifications "active" and "inactive," or *alpha* and *beta*.

(2) That all "primary" amorphous carbon consists essentially of a stabilized complex of hydrocarbons, adsorbed on a base of "active" or *alpha* carbon.

We will discuss these primary postulates in order.

The active modification of carbon is characterized by a high specific adsorptive capacity for gases, etc. The inactive modification carbon exhibits no special adsorbtive capacity whatever.

In addition to this distinction in specific adsorbing power, active and inactive carbon differ in two other important particulars, *i. e.*:

(1) Temperature of formation.

(2) Chemical activity, or susceptibility to oxidation.

The active modification is formed whenever carbon is deposited at relatively low temperatures by chemical or thermal decomposition of carbon-bearing materials; in general below 500 to 600° C.

The inactive modification results from similar decomposition at higher temperatures, in general above 600 to 700° C.

The active form is rapidly attacked by oxidizing agents. Dr. Hulett has shown that slow oxidation occurs at room temperatures. The inactive form is relatively stable toward oxidizing agents, resembling graphite in this particular.

It would be premature to assert that these two forms of carbon are true allotropic modifications. It is not yet established that both forms are amorphous. Attempts to find a transition temperature, or to convert *alpha* into *beta* carbon, have been tried without definite success, at temperatures up to 1,600° C.

This much is established. The two forms are characteristically distinct and easily differentiated, both by their properties and conditions of formation.

Hydrocarbons such as methane, ethylene, benzene, etc., the decomposition points of which are normally above 700° C., in general give only inactive carbon. Hydrocarbons such as acetylene, which may be cracked at temperatures as low as 300° C., may give active carbon at lower temperatures and inactive carbon at higher temperatures. The temperature at which molecular carbon is set free is apparently the controlling factor in determining whether it is of the active or inactive variety.

The second part of the general theory relates as stated above to the definition of primary carbon as a stable adsorption complex of active carbon and residual hydrocarbons.

In the ordinary process of distillation of carbonaceous materials, at relatively low temperatures active carbon is first formed by the thermal decomposition of unstable hydrocarbons. By virtue of its specific adsorptive properties this active carbon adsorbs a further quantity of hydrocarbons, and these hydrocarbons are thereby stabilized to a remarkable extent, so that they are retained under conditions of temperature, pressure, etc., at which they would otherwise be quickly eliminated as such, or suffer decomposition.

For example quantities of a hydrocarbon resembling anthracene, the boiling point of which is 360° C., have been isolated from cedar charcoal which had been previously calcined to 850° C. In the same manner chlorine substitution products of hydrocarbons may be obtained in quantity by passing chlorine gas at

high temperatures over cocoanut or other charcoals which have been calcined to similar temperatures. This illustrates the power of active carbon to stabilize its adsorbed materials.

This stabilized complex of hydrocarbons adsorbed on a base of active carbon is the product which we have defined as primary carbon because it is the original product first occurring in low temperature distillation of carboniferous materials. Many commercial grades of animal and vegetable charcoal are of this character; as are also such cokes as result from the low temperature distillation of bituminous coals, mineral bitumens, etc.

Anthracite coals may be regarded as native primary carbons, since they have been shown to possess an active carbon base saturated with adsorbed hydrocarbons.

Bituminous coals may be similarly regarded, although their hydrocarbon content is in great excess of the adsorptive capacity of the active carbon base. Such excess may be readily removed by distillation under proper conditions.

Ordinary coke, on the other hand, is heavily charged with inactive carbon resulting from the gas treatment to which it has been subjected, *i. e.*, the decomposition in it of volatile hydrocarbons at high temperatures.

Sudden heating of primary carbons to very high temperatures results in the decomposition of the adsorption complex, and the sudden liberation of its hydrocarbons at temperatures much above their normal decomposition point. The latter then break down forming inactive carbon deposits upon the active carbon. In this way a primary carbon may become "self gas-treated."

Thus the carbon products resulting from the "gas treating" of primary carbon at high temperatures by volatile hydrocarbons, either self contained, or introduced from without, may be properly termed secondary carbon, which thus possesses a variable composition of active carbon, hydrocarbons and inactive carbon, depending upon the extent and conditions of the gas treatment.

Primary carbon is therefore essentially an adsorption complex of hydrocarbons adsorbed by a base of active carbon, with or without associated non-adsorbed hydrocarbons.

Secondary carbon is primary carbon partially or completely broken up with the formation or introduction of inactive carbon, by the high temperature decomposition of its hydrocarbons.

From the above description it is self evident that primary carbons as such will not possess a high specific adsorptive power, the active carbon being saturated by the hydrocarbons adsorbed by it. The process of activation must therefore consist fundamentally in the separation and removal of the hydrocarbons from the active carbon.

The correctness of the general principles just outlined has been substantiated in a very interesting manner. According to theory carbon deposited at low temperatures and absolutely free from hydrocarbons, should be the purest form of active carbon and require no activating process. Therefore carbon was prepared from the reaction $2CO = C + CO_2$, at 300° C., using ferric oxide as a catalyst.

The carbon so formed upon removal of the iron proved to have a specific adsorptive capacity appreciably greater than the best samples of active carbon prepared from hydrocarbon sources.

The practical elimination of the hydrocarbons from primary carbon is, however, extremely difficult.

Because of the stability of the adsorption complex, the temperatures required to accomplish this thermally are so high that secondary formation of inactive carbon by self gas treating is inevitable. Prolonged heating for days under carefully controlled conditions will partially accomplish the desired object, yielding an impaired and gas treated product, which contains a small amount of active carbon. To secure even partial success with this method requires specially selected and prepared forms of primary carbon, in which the character of the hydrocarbon content is such that either the stability of the adsorption complex is lessened or the tendency to gas treating is a minimum.

Having independently discovered the essential principles of this method, it was abandoned because of its practical limitations. It was later found that the British and French had developed this method to the commercial stage.

The second general method of activation suggested by the theory is to attack the primary carbon by some form of selective chemical action.

The most successful of these is differential oxidation. It has been shown that the adsorption complex could be broken down

and the hydrocarbons oxidized or partially decomposed and distilled away from the active carbon.

While many forms of oxidation, both wet and dry, gave some degree of separation, and consequent activation, the yield and quality of activated carbon obtained varied greatly with the kind of oxidizer employed and the manner of its control.

Since inactive carbon is highly resistant to oxidation it is essential to the proper practice of activation by controlled oxidation, that the formation of inactive carbon be avoided at every stage of the operation, including the production of the primary carbon itself. At the same time considerable latitude is permissible for the reason that the tendency to gas treatment varies with the chemical nature of the adsorbed hydrocarbons, *i. e.*, with their decomposition points.

Hydrocarbons present in bituminous coals are found to deposit inactive carbon more readily than hydrocarbons derived from certain other carbon complexes such as may be prepared from certain woods and nuts. Hence it is that the adsorptive carbon of commerce was hitherto produced only from the latter sources, and not by reason of any inherent impossibility of preparing active carbon from coals, petroleum, and other mineral deposits.

The commercial developments of the oxidation principle as worked out by us and adopted by the Chemical Warfare Service, consisted in the air process, the steam process, and later the use of CO_2. A modification involving a combination of steam and CO_2 by enriching flue gases with steam was developed with considerable success by the Development Division, C. W. S., at Nela Park.

The discovery of the availability of anthracite and natural coals, and the possibility of manufacturing synthetic primary carbon from pulverized materials by the use of the usual binders employed in carbon manufacture, saved us from a critical shortage of nut materials in the later months of the war.

Certain special forms of activated carbon arising from these developments were known by the Gas Defense as Dorsite made from cocoanut, Bachite made from anthracite, and Carbonite a synthetic product made by carbon manufacturing processes, from lampblack, powdered coal, or other suitable materials.

The underlying principles of the theory as to the nature of

primary carbon are strongly confirmed by the fact that two entirely different methods of activation suggested by it, *i. e.*, controlled distillation, and differential oxidation, are each effective, though in very different degrees.

A modification of the second method is also suggested by the theory, which would consist in the addition of chemical reagents to act catalytically or directly during the primary carbonization to break down the stabilized hydrocarbons at temperatures below the critical range in which inactive carbon is formed.

This method was successfully employed by us to duplicate German war-charcoal, thus indicating the method by which the latter was formed, which consisted in impregnating wood with various metal salts before carbonization, and later extracting with acid. This was afterwards verified by the Army Commission visiting Germany.

The amount of active carbon formed by this method is very small in comparison with the better methods of selective oxidation. On the other hand, the extraction of the dispersed inorganic material leaves a very fine and effective capillary structure capable of adsorbing large quantities of gas at high concentrations of the latter. In this connection it is necessary to explain that we have clearly shown the possibility of differentiating between two types of adsorptive capacity, one due entirely to the arrangement and physical structures of the adsorbent, which we term the capillary capacity, and a second type characteristic of the material of which the adsorbent is constituted. Capillary capacity is distinguished by large capacity at high gas concentrations and by rapid equilibrium between adsorbent and gas phase, in either direction, the gas being removed as readily as it is adsorbed. Specific capacity is characterized by very considerable capacity at low gas concentrations, and by extremely low rate of loss during reversal of the adsorption process.

Thus by saturating an adsorbent and then noting the rate at which different portions of the adsorbed gas are removed, it has been found possible to evaluate the specific and capillary components of the total adsorptive capacity.

The weight of gas retained, after rapid weight loss has ceased, called the "retentivity" of the adsorbent, is a measure, therefore, of the proportion of active carbon present, in a given weight of

adsorbent. This test applied to pre-war charcoals shows that their adsorptive capacity is almost wholly capillary and dependent upon the physical structure left by distillation in the primary carbon. In general even this capacity is extremely small compared with present standards.

German charcoal is distinguished by the very high ratio of capillary to specific capacity. On the standard accelerated tube tests employed by the Chemical Warfare Service German charcoal thus showed up very well because of the high concentrations of gas employed. If one were to breathe through the coal at the end of such a test, however, one would soon inhale 75 percent, or so, of the gas previously adsorbed. Thus German charcoal received for a time a wholly unwarranted reputation.

We have now outlined a general theory of the nature of amorphous carbon and the principles underlying the successful isolation of active carbon.

As a working hypothesis the conceptions above outlined have proved their worth by enabling us to develop methods for the commercial manufacture of adsorbent and catalytic carbon, of a character unapproached in pre-war charcoals and from a variety of raw materials hitherto considered useless for such purposes.

Upon the chemical side we have provided a new means of attack upon the problems of the structure and constitution of the various forms of amorphous carbon.

The actual development of the two principal conceptions underlying the complete theory was the reverse of the order of exposition just given. At the outset of the work the fundamental question was w h y and h o w adsorbent charcoals differed from other charcoals less adsorbent.

Because of the vital urgency of the problem at that time and the need for haste, we knew that it would be fatal to back a wrong hypothesis. So far as we could learn the following hypotheses were current:

(1) That a special form of hydrocarbon film was left under certain conditions of distillation which was especially active.

In the case of such gases as chlorine which may form substitution products, such a possibility certainly exists even in the light of present developments.

(2) That adsorption depended upon the physical structure of the adsorbent, *i. e.,* the diameter of the capillary pores.

As we have seen this actually represents the type of adsorption previously common in adsorbent materials, other than active carbon, but is not the type of specific adsorption desired for effective field protection against low concentrations of toxic gases.

(3) That adsorptive power depended upon the quantity of fixed gases as carbon monoxide, oxygen, etc.

The difficulty with the latter view was that, if true, it suggested no line of attack.

After considering the above we adopted the hypothesis that absolutely pure amorphous carbon would be active, and that the most obvious impurities in charcoals formed by destructive distillation were hydrocarbons.

It was by no means readily accepted by many chemists that ordinary low boiling hydrocarbons could exist as such in chars which had been heated up to nearly 1,000° C. for hours.

The crucial experiment was therefore the proof that ordinary hydrocarbons resembling anthracene for example could be removed as such from a charcoal previously calcined to 850° C. by simply distilling it in the presence of air at 300° C.

This not only proved the existence of the hydrocarbons, but indicated directly that by oxidation they could be broken down from the adsorption complex. No explanation was forthcoming, however, as to why certain forms of carbon could be activated in this manner, while others could not.

It was not until work was commenced on coals and artificial carbons that the significance of the decomposition temperature of the carbon was discovered.

This clue cleared up the entire field and provided the explanation for many previous anomalies, as why, for example, artificial carbons taken from the same furnace could, some of them, be activated and others not. Our observation that all forms of retort cokes were wholly inactive and incapable of activation was also explained. The fundamental conditions surrounding the formation of primary carbon, if the latter was to be capable of activation, were thus completely defined.

To sum up, active carbon is essentially a special form of pure amorphous carbon, deposited at low temperatures and free (1) from the adsorbed, stabilized, hydrocarbons which are normally associated with it and which lessen its power of combining with other substances; (2) from inactive carbon formed by gas treating, *i. e.*, by the decomposition of hydrocarbons upon its surface at high temperatures.

It is possible for the first time to prepare intelligently such active carbon on a large scale from whatever sources of carbon-bearing material may be cheapest, or most desirable for the particular purposes in view.

The unique properties of this material both as catalyst and adsorbent suggest that it will find many industrial applications.

ACKNOWLEDGMENTS.

This paper is a resumé of the general principles of carbon activation as originated and developed by the writer, during investigations carried out at the Government's request in the laboratories of this company. The purpose was to obtain an absorbent carbon capable of affording adequate defense against certain toxic gases employed in warfare, for which no chemical protection existed. That these theories and the commercial processes resulting therefrom met with such rapid development and signal success in the production of American gas mask carbon, is due to the self-sacrificing co-operation of many workers whom it is impossible to mention individually.

Special acknowledgment is due, however to Mr. H. D. Batchelor, the laboratory director, and the members of the research staff, whose splendid support and co-operation were unfailing; to Dr. W. K. Lewis and Lieutenant-Colonel Arthur B. Lamb, of the Research Division of the Chemical Warfare Service, for their unceasing moral and material support; and also to the fine body of chemists and engineers constituting the charcoal research unit, National Carbon Company Detachment, of the Chemical Warfare Service, who devoted themselves to the work with untiring loyalty and notable success.

Acknowledgment is also due to Colonel F. M. Dorsey, Development Division, C. W. S., whose prompt acceptance and energetic

engineering development of our processes, was a vital factor in the rapidity with which production for the army was achieved.

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National Carbon Co., Inc.,
Cleveland, Ohio.

DISCUSSION.

W. D. BANCROFT[1]: I should like to ask Dr. Chaney one or two points: first, what he considers to be the density of the active carbon, the true density? I understand that Mr. Hulett has shown that the true density of almost all these carbons is somewhere in the neighborhood of 1.8. And then, I would like to ask him what he considers the true density of the active carbon, which I assume is higher; and then the question would come up, what is the relation between active charcoal, inactive charcoal and graphite; that is, whether inactive charcoal is really a form of graphite which you have in the very thin film on the surface of the active carbon, and which therefore does not give you the ordinary properties of graphite in mass? I do not imagine that Dr. Chaney has any final theories to give in regard to that, but he may have some points of view which will help. Then there is one more point: he has cited here his discovery that hydrocarbons which boil at a relatively moderate temperature are still held by the charcoal at temperatures even of a thousand degrees, due to adsorption. In some of the work with poison gases, it was shown that you get similar phenomena, only not quite so marked, in the case of liquids; that is, if you take active charcoal and treat it with benzene or other organic liquids, the charcoal can be heated 100° above the boiling point of the liquid, and on cooling down there will still be enough of that liquid left in the charcoal to affect very materially the rate of adsorption of chloropicrin by charcoal; in other words, distinct amounts of volatile liquid are retained by charcoal even when heated at least 100° above the normal boiling point of those liquids. You have here

[1] Professor of Physical Chemistry, Cornell University.

precisely similar phenomena, except that you are dealing with a volatile liquid instead of a substance that is solid at the ordinary temperature.

F. C. FRARY[2]: It seems to me that the difference which this paper points out between the two forms of active carbon is extremely important as clarifying the use of active carbon in catalytic processes. Mr. Chaney points out that the active carbon of the capillary type comes very quickly into equilibrium with gases which are in high concentration, and equilibrium takes place from all sides; that is, no matter which gas you have in excess, the equilibrium is very rapidly established, while in the other type, which is more particularly produced by the processes used for making gas masks, the charcoal takes up material but gives it out very slowly. Now for a catalyst, which is designed to be used in making two gases combine with each other, for instance the combination of carbon monoxide with chlorine, such as we were particularly interested in during the war, I call attention to the fact that we prefer the capillary type, and that the other type actually makes trouble. I can give you an illustration of that from an experience which we were not able to explain at the time, but which is perfectly explained by Mr. Chaney's theory. In carrying on this reaction on a large scale, we of course attempted to mix carbon monoxide and chlorine in as nearly as possible theoretical proportions, using a slight excess of carbon monoxide in order to be sure that a minimum of free chlorine existed in the resulting gases. Occasionally something would go wrong and our free chlorine concentration for a little while, say four or five minutes, might run up to several percent. We found then that it was entirely insufficient to change the excess of carbon monoxide to three or four times what it would normally require to get a fully converted gas; we would still get chlorine, even with three or four times the normal excess of carbon monoxide. The only thing to do was to shut the chlorine off entirely, purge with CO, and start up again. In other words, this form of active carbon which was desired for gas masks was making us trouble, because it took up the chlorine and would not come to equilibrium

[2] Director of Research, Aluminum Co. of America, New Kensington, Pa.

with the carbon monoxide but continued giving off chlorine in spite of the fact that we had an unusually large excess of monoxide there. I would point out that the kind of carbon which is most apt to give satisfactory results for a catalyzing reaction between two gases is the capillary type and not the gas-mask type. As a matter of fact, we tried gas-mask charcoal in the early days to effect that reaction, and we found that the charcoal was extremely active for about three minutes, until it was itself saturated with the product of the reaction, and then it would not do anything at all: it was dead. That is all very nicely explained by Dr. Chaney's present theory. I feel that this paper is a very great contribution to our knowledge of the theory of the use of carbon as a catalyst.

W. D. Bancroft: I do not want to anticipate the symposium on catalysis, but I should like to point out that Mr. Frary's statement is probably a little too broad; that is, his statement about the difference between the two charcoals is undoubtedly correct for the case with which he was working, where the adsorption was very marked. On the other hand, it seems to me very probable that if you are using charcoal as a catalytic agent in cases of substances which are not adsorbed very strongly by charcoal, you will find that Mr. Chaney's active charcoal is a much better catalytic agent than the other charcoal which worked more satisfactorily in the case of the manufacture of phosgene. It seems to me that it is probable you have to work out in each case a certain relation between the degree of adsorption and the catalytic action, and I think it would be a great pity to have the thing accepted quite in the form that Mr. Frary put it.

A. Taylor: I would like Mr. Chaney to give us some amplification of the two types of absorption which are being discussed today. What is his idea concerning the nature of capillary absorption? And what is his idea of the nature of the other type of absorption? What I wanted to ask that for is this: does one get capillary absorption according to the Sigmond theory as a liquid content of the capillary, and if so, does that take place above the critical temperature of the gases employed? That is a very important point, which apparently has not yet been discussed in connection with the capillary theory of absorption.

S. A. TUCKER[3]: We have not had an answer as to the specific gravity of these different forms of carbon. I am interested in that also. I noticed several years ago in experiments with granular coke, which is used as a resistor in the electric furnace, that you only get rid of those volatile hydrocarbons, as I thought of them at the time, at very high temperature. Now it seems to me that it must be in the neighborhood of 1600° or 1700° C. before you are really free from such constituents. This is shown by the fact that taking ordinary gas coke and heating it, the resistance falls steadily with rising temperature, and it takes both the element of time and temperature to get rid of volatile constituents. I suppose that is in line with what we have just been discussing.

W. D. HARKINS[4]: I simply want to call attention to the fact that Mr. Ewing, who has been working under my direction, has been studying the densities of charcoals, as spoken of by President Bancroft, and we find that the density, at least the calculated density, will depend greatly upon what theory is used in interpreting the experimental results. For example, we find that the density of one charcoal varies from 1.863 to 2.094, depending upon what liquid is used in the determination of the densities. The density increases as the compressibility of the liquid used in determining density increases. The only theory that seems at all satisfactory in this connection is that the liquid which is absorbed by the charcoal is very highly compressed. We used some charcoals which were very kindly sent us by Dr. Chaney, and found that they give determinations similar to the cocoanut charcoals.

W. D. BANCROFT: All these things lead off to entirely different cases. I should like to bring up one point that perhaps Mr. Harkins might study, which does not come strictly under electrochemistry, but is suggested by it. He has pointed out that you get different apparent densities of charcoal depending upon the nature of the liquid you use, which I assume is due to the fact that you get different degrees of adsorption and condensation of the liquid on the surface of the charcoal. That suggests a line of attack which would certainly be very interesting to those of us who are

[3] Chemical Foundation, Inc., New York City.
[4] Kent Chemical Lab., University of Chicago, Chicago, Ill.

concerned with colloid chemistry. It was shown that the degree of adsorption of iodine from different liquids did not follow the same order with different charcoals. Now that fact is absolutely impossible unless you have an adsorption of the liquid by the charcoal, which was not taken into account in the experiment, and the question has been, how would you test this in some way so as to come out with results that would be more or less quantitative? I have not, myself, seen any very good way of attacking the problem, but it seems to me now that if Mr. Harkins will take those same solvents and those same types of charcoal and will determine the apparent densities of the charcoals in those different solvents, we can then arrange them in an order in reference to the liquids, which might then clear up the question of the relative adsorption of the iodine. That would certainly help us out of a very awkward theoretical situation in which we now are and for which no satisfactory explanation has been offered.

COLIN G. FINK[5]: Assume you have a case of iodine and a special solvent: without the iodine the solvent is strongly absorbed and in the presence of the iodine it might not be absorbed at all; or *vice versa*.

W. D. HARKINS: I might say that we expect to do further work upon this subject. There might be some question as to whether it is a time effect or not, but it seems probable that it is not. The theory of which I spoke as being the only satisfactory one at present is not the only one that we might advance, and we will consider the other theories when we publish our work on the subject.

H. H. SHELDON[6]: Mr. N. K. Chaney stated that the general theory in its complete form rests upon two postulates, one of which is: "that elementary carbon (other than diamond and graphite) exists in two modifications, 'active' and 'inactive' or *alpha* and *beta*."

It would seem from data obtained here that the definitions of active and inactive would need to be modified before this classification can have any meaning, since charcoal can be made which

[5] Head of Laboratories, Chile Exploration Co., New York City.

[6] University of Chicago. This discussion originally appeared in "Science" (1919), 50, 568.

is the reverse of other charcoals in that it is relatively more active for hydrogen than for nitrogen as shown by the following data:

Charcoal	Initial Volume	Volume of Hydrogen Adsorbed	Volume of Nitrogen Adsorbed
Usual type	926 cc	914 cc.	926 cc.*
Usual type	1,780 cc	1,657 cc.	1,780 cc.
New Sample 1.....	926 cc	907 cc.	666 cc.
New Sample 2	926 cc	900 cc.	755 cc
New Sample 3	926 cc	874 cc.	406 cc.

* Not saturated in this particular case.

Each of the volume measurements given were calculated from pressure readings and are reduced to normal pressure and temperature. The amount of charcoal used in each case was 25.7 g. and this was left at liquid air temperature until saturated. The gases were used separately and not as mixtures.

The difference in treatment of the last three samples was slight, yet sample 1 shows figures lying on the outside of those for sample 2, *i. e.*, the figures of sample 1 have approached each other for sample 2. Much more striking samples can no doubt be prepared.

A report of this work will be published when completed, but this will serve to point out an apparent incompleteness in the theory set forth by A. B. Lamb[7] and by N. K. Chaney.

The author is indebted to Dr. H. B. Lemon for valuable advice and assistance in this work.

N. K. CHANEY: Dr. Bancroft has raised a number of interesting points, upon which we are now at work but are not prepared to answer just yet. We hope to be able to determine ultimately the physical basis of the differences between the "active" and "inactive" forms of carbon; whether these are, as we believe, two distinct allotropic modifications, or merely a single modification exhibiting a wide and indeterminate variation in properties. We have evidence, which it would be premature to discuss here, suggesting that *"beta"* carbon is amorphous graphite. All of the known forms of graphite are really crystalline, even the so-called "amorphous graphites."

[7] J. Ind. and Eng. Chem. (1919), **11**, 420-467.

Dr. Bancroft's question as to the real density of active carbon raises a debatable question, upon which Dr. Harkins' work promises to shed a good deal of light.

Determining the real density of activated carbon in the ordinary routine way by evacuating at 40° C. in kerosene of known density, we found that the real density apparently increased with the degree of activation, approaching the values of graphite, about 2.2. Dr. Hulett, working with much more refined methods, evacuating at 300°–400° C. and using water as the liquid, found much lower values, about 1.84. The puzzling thing, which neither of us could explain, is that the special refinements employed by Dr. Hulett would naturally be expected to give h i g h e r values, since the error inherent in such a method is the failure to remove the gases completely and to secure complete penetration of the pores of the charcoal by the liquid.

Dr. Harkins obtains values with water similar to those obtained by Dr. Hulett, but with benzene and similar liquids he obtains the high values corresponding to those that we found with kerosene. This discrepancy is explained by the compressibility theory, as is also our observation of the regularly increasing densities with increasing degree of activation. On the other hand, recent work by Dr. Hulett does not check the values found by Dr. Harkins with the same liquids. Dr. Hulett finds lower values as a rule, but also finds "drift," or increasing density with time and pressure. Why Dr. Harkins should obtain maximum values toward which Dr. Hulett tends to approach, is not yet satisfactorily explained.

With regard to Dr. Frary's discussion of catalysis, I think it true that we have here a very significant clue as to catalytic behavior. But I agree with Dr. Bancroft that it is not quite as simple as might at first sight appear.

The "capillary" type of adsorption, I believe, has no direct influence upon catalysis except as it favors increased surface and diffusion. A good example of this is Dr. Patrick's silica gel, which shows only "capillary" adsorption and has no "specific" adsorptive power or "retentivity" at all, and exhibits no catalytic activity. Most pre-war commercial charcoals fall in the same category. In the case of the phosgene reaction cited by Dr. Frary the reaction seems to depend upon the maintenance of an adsorp-

tion layer of CO, which then reacts with the gaseous chlorine. If the reverse of this is permitted, and the adsorbed layer consists of c h l o r i n e, then reaction stops.

If the "specific" adsorptive power of "active" carbon is due to a field of force arising from the presence of unsaturated carbon valences incident to its ultimate molecular arrangement, then the

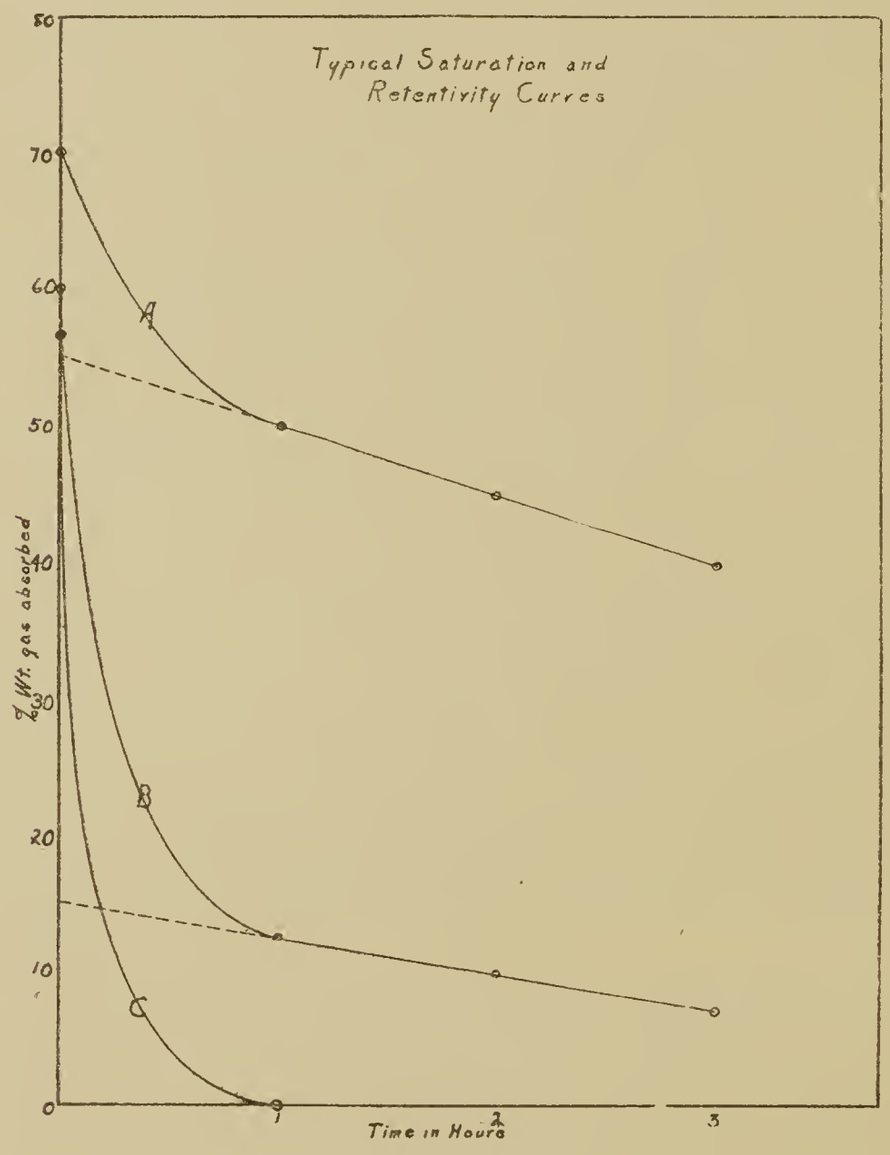

FIG. 1.

average intensity of such a field can be modified by the amount of "concentration" of active carbon present in the adsorbent surface.

If the adsorbed gases are themselves also unsaturated in character, specific differences in their adsorption behavior may appear, quite apart from the general relations predicted from their relative boiling points, etc.

In such cases as the phosgene reaction, it may well be that the field of force must be adjusted within rather delicate limits to prevent a reversal in the adsorption layer, despite the advantage given the CO by being first on the ground and in high effective concentration (the concentration of free chlorine being normally kept down by its reaction with the CO layer).

The proper balance of modification of "specific" adsorptive power, rather than its absence, would seem to offer the best clue to a study of the rôle of adsorption phenomena in catalysis.

For certain gas reactions, active carbon made from anthracite coal which has both a relatively low capillary capacity and a low specific adsorptive power shows very superior catalytic activity.

Mr. A. Taylor has asked me to define more sharply the distinction between "specific" and "capillary" capacity. The experimental difference is well marked. The simplest theoretical assumption is that capillary adsorption is the filling of actual cells with liquid due to lowered surface tension, and dependent upon the size of the capillaries, *i. e.,* the physical structure of the adsorbent only. This is the type of adsorption occurring exclusively in Patrick's silica gel, and would not take place above the critical temperature of the gases adsorbed.

The specific capacity has been assumed to represent a field of force—probably due to unsaturated valences—which is independent of a grosser physical structure of the adsorbent; *i. e.,* it represents adsorption on a plane surface. It differs in magnitude, and may possibly be effective to some extent above the critical temperature. The experimental difference is best illustrated by the following curves. (See Fig. 1.) Three typical charcoals are shown in which the values at the origin of the curves represent complete saturation at a given temperature with a given partial pressure of gas. The gas adsorbed is expressed as a percentage of the weight of carbon employed. The time curves show the percentage weight of gas left in at definite intervals during evacuation at 100° C.

There is a rapid loss at first, after which under standard conditions the residual weights lie on a practically straight line. We have arbitrarily extrapolated this straight line back to the point of origin (see dotted lines) and accepted the values on the y-axis so obtained as a relative measure of the "specific" capacity. The

difference between the straight line and curved line intercepts on the y-axis thus represent the "capillary" capacity, under the particular conditions of saturation employed.

Curves *A* and *B* show about the same order of total saturation capacity. But *A* has a high retentivity and slight capillarity, while *B* has a low retentivity but high capillarity. *C* represents the type of material having capillary capacity only, such as unactivated charcoal.

Mr. Sheldon's observations are of great interest and their proper explanation may prove of considerable theoretical value. That my present theory is "incomplete" in that it fails to explain these phenomena, is readily granted. It does not seem equally obvious, however, that the characteristic distinctions already observed between "active" and "inactive" carbon lose any meaning which they may now have as the result of such uninterpreted observations.

If Mr. Sheldon believes his data establish beyond question a q u a l i t a t i v e difference in the nature of the field of force exerted by "active" carbon as such, I cannot agree with him. That the manifestation of it, as shown in apparent adsorptive power, is capable of modification by other factors is hardly to be doubted.

Early in the work of the Chemical Warfare Service similar anomalies were reported with respect to various toxic gases, and the view was held by prominent workers that each gas presented a wholly specific problem, and that no general method which would improve carbon for all types of gas adsorption was likely. I held the contrary view, that the primary problem was the improvement of the adsorptive power *per se,* after which come special modifications to meet specific requirements of individual gases. The explanation of the anomalies observed were found to be of three kinds:

(1) The distinction between "capillary" and "specific" adsorption already noted. In the "standard tube test" methods employed by the C. W. S. (see Jour. Indus. Eng. Chem. (1919), **11**, 519) variable percentages of the "capillary" capacity of a charcoal are effective depending upon the height of layer, rate of flow of gas, gas concentration, etc. Moreover, as between different gases, the capillary capacity is relatively much more effective for high boiling liquids than for low. Since the "capillary" capacity

bears no fixed relation whatever to the specific capacity, variability in the ratio of the two gives very different results with different gases. For example, it was claimed that continued "activation" or oxidation which improved a charcoal for chlorpicrin, impaired it for phosgene. A careful comparison with a given charcoal, activated to different degrees, showed that in a certain range of oxidation the chlorpicrin adsorption increased 100 percent, while the phosphene adsorption increased but 10 percent. It was found in this same range that the "capillary" capacity was enormously increased, whereas the "retentivity" remained constant. It was further found that in this particular case the phosgene was completely hydrolyzed to CO_2 and HCl, of which the latter only was adsorbed. Thus the difference was due to the much greater effectiveness of the capillaries for chlorpicrin than for the more volatile HCl, the latter being held mainly by "specific" adsorption.

(2) The presence of a film of a third substance in which two gases were differently soluble. The adsorption of HCl for example is enormously increased by a water film.

(3) Variability in the rate of catalytic decompositions, whereby the nature of the adsorption products is changed. This is illustrated by the phosgene reaction above. Under certain cases phosgene is adsorbed, under others the more difficultly retained HCl.

Speculations as to the cause of the reversal in adsorptive capacity of Mr. Sheldon's charcoal samples for H_2 and N_2 are not justified by the data he presents, except to point out that various possibilities exist which would in no wise vitiate the significance of the distinctions already observed between *"alpha"* and *"beta"* carbon. It is noticeable that adsorption for both H_2 and N_2 is progressively impaired, which suggests the possibility that a change has taken place which affects the diffusion of the N_2 molecule more than that of the H_2 molecule. This might result either from a constriction or superficial closing of the smallest pores, or from an adsorption layer of some other kind.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

THE PENETRATION OF IRON BY HYDROGEN.*

By T. S. Fuller.†

Abstract.

The results are given of experiments showing the effect of various conditions on the penetration of iron by nascent hydrogen at temperatures from 20° to 100° C. Hydrogen was generated on the outside of an iron tube by immersing the tube in a solution, and the volume of gas penetrating to the inside of the tube was measured. The rate was greater for a unit immersed without electrical connections than when the unit was used as a cathode, but in the latter case the greater the current the greater the penetration. The rate also increases with the temperature. Copper is not penetrated, but a coating of tin on the iron increases the rate. The effects of other conditions are shown.

Some time ago the writer published the results of some plating experiments on steel springs[1] in which he concluded—"The facts are all in accord with the assumption that the absorption by the steel of atomic or nascent hydrogen liberated at the cathode is the cause of the embrittlement of steel springs in the plating-bath." The results of these experiments, together with those of Charpy and Bonnerot,[2] Coulson,[3] Merica,[4] and others, lead to the study of the conditions which govern the penetration of iron by atomic hydrogen, namely current, treatment of the iron, temperature, electrolyte and surface coatings.

* Manuscript received July 30, 1919.

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[1] Trans. Am. Electrochem. Soc. (1917), **32**, 247.

[2] Compt. Rend. (1912), **154**, 592; (1913), **156**, 394.

[3] Trans. Am. Electrochem. Soc. (1917), **32**, 237.

[4] Met. and Chem. Eng. (1917), **16**, 496.

It is well known that iron at room temperatures is impermeable to gaseous or molecular hydrogen (H_2), but that at higher temperatures it becomes more or less permeable. It is not so well known, perhaps, that iron at room temperature is permeable to nascent or atomic hydrogen (H). These problems have been investigated by Charpy and Bonnerot.[5] Their apparatus consisted of a steel tube 0.5 mm. thick, connected to a pump which maintained a constant pressure of 0.2 mm. inside the tube. This tube was placed inside another one of porcelain, which was heated, and through which gaseous hydrogen at atmospheric pressure flowed, surrounding the steel tube. The iron was found to be impermeable up to a temperature of 325° C. The following table, taken from Charpy and Bonnerot's data, shows the amount of gaseous hydrogen expressed in cubic centimeters per hour, collected in the steel tube:

Temp.	Cc. of H_2 per Hour
350°C.	1.1
450°C.	3.2
550°C.	8.5
750°C.	30.0
850°C.	42.0

Pressures as high as 26 atmospheres were observed by these writers in an iron container which was made cathode in acid solution.

APPARATUS.

A very convenient as well as novel form of apparatus has been developed and used for the experiments described in this paper.

It consists of a seamless iron tube plugged at the bottom, and sealed at the top to a glass U tube, having one arm closed and calibrated, and the other open. After assembling, the apparatus is completely filled with mercury, and when in operation hydrogen penetrates the iron tube, and quickly rises and displaces the mercury in the closed and calibrated arm of the U. Fig. 1 shows the construction of one of the units. *A* is a section of seamless iron tubing 12 inches (30.5 cm.) long, and 11/16 inch (17.5 mm.)

[5] *Loc. cit.*

outside diameter, with a wall 1/16 inch (1.6 mm.) thick. *B* is a cone-shaped tube of nickel steel having its small end brazed to the iron tube *A*. The large end of *B* makes a vacuum-tight seal with the bottom of the glass tube *C*, whch terminates at the top in the U tube *D-E*. *D* is closed at the top and calibrated, and *E* is open. The capacity of *D* is about two cubic centimeters. *F* is a platinum wire which is sealed through the glass and dips into the mercury. *G* is an iron plug which is brazed into the bottom of the iron tube *A*. *H* is a section of rubber tubing which protects the brazed joint between *A* and *B*, as well as *B* itself, from the action of the electrolyte, and *I* is a coating of rubber cement which protects the brazed joint between *A* and *G*. The finished unit is so arranged that the electrolyte comes in contact with only

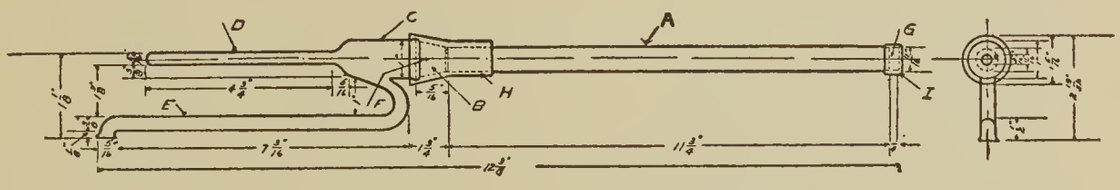

FIG. 1. Apparatus for Measuring the Rate of Hydrogen Penetration in Iron.

iron and glass. When the unit is complete it is sealed to a vacuum pump and tested for leaks. None have been used which could not be pumped out to a pressure of 0.005 mm. of mercury without showing a leak. After filling with mercury the units are ready for use.

Fig. 2 shows one of the units completely filled with mercury, and Fig. 3 another unit, the mercury in the measuring tube of which has been partially displaced by hydrogen which has collected. Fig. 4 shows the scheme for measuring the rate of hydrogen penetration at different temperatures. Six units are immersed in glass tubes, containing the electrolyte, these tubes being in turn immersed in an electrically heated water-bath.

EFFECT OF CURRENT.

To determine the relation between current and the rate of hydrogen penetration, four units were made cathode in 1 percent sulphuric acid at room temperature, the units being connected in the circuit by means of the platinum wire *F*. Platinum anodes were used. Two of the units were electrolyzed with a current

of 0.2 amp. and two with a current of 0.5 amp. The potential across the former cell was 2.5 volts and across the latter 2.8 volts.

Two of the units were electrolyzed a second time.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
(1) Iron—New.....	0.2 amp.	19 hrs.	20°C.	0.55 cc.
(2) Iron—New.....	0.5 "	" "	"	1.85 "
(3) Iron—New.....	0.2 "	24 "	"	0.15 "
		39 "	"	0.90 "
		42 "	"	1.00 "
(4) Iron—New.....	0.5 "	24 "	"	0.40 "
		39 "	"	1.35 "
		42 "	"	1.50 "
(5) Iron—Same unit as used in (3).	0.2 "	24 "	"	0.30 "
		33 "	"	1.00 "
		48 "	"	2.40 "
(6) Iron—Same unit as used in (4).	0.5 "	24 "	"	0.30 "
		33 "	"	1.47 "
		48 "	"	2.40 "

In every case a current of 0.5 amp. allowed more hydrogen to penetrate the iron than did a current of 0.2 amp., but not two and a half times as much. The velocity of hydrogen penetration is not a straight line function of the current. The rate is influenced by the current—the higher the current, at least for such densities as were used in this experiment, the greater the velocity of penetration.

NO ELECTRICAL CONNECTIONS.

One of the units was immersed, without electrical connections, in a solution of one percent sulphuric acid, in other words "pickled."

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—New	24 hrs.	20° C.	0 cc.
	27 "	"	0.02 "
	33 "	"	0.42 "
	48 "	"	2.00 "

At the end of 48 hours, hydrogen somewhat in excess of 2 cc. had collected. This rate of penetration is higher than the rate for the units made cathode with currents of 0.2 or 0.5 ampere.

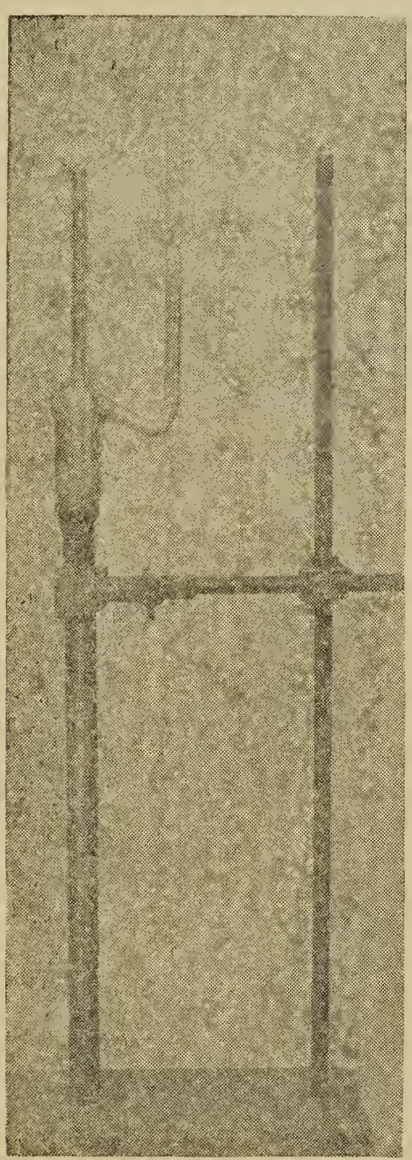

FIG. 2. Measuring Tube Completely Filled with Mercury.

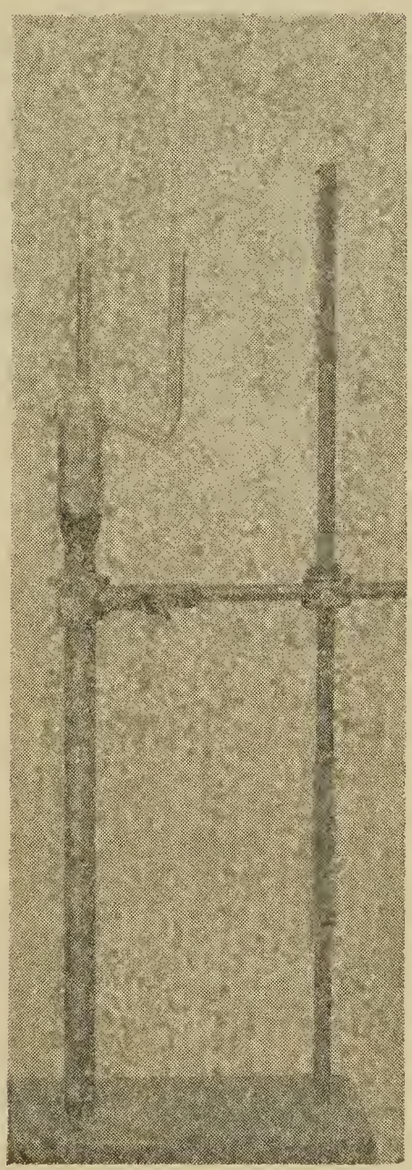

FIG. 3. Mercury in the Measuring Tube Partially Displaced by Hydrogen.

EFFECT OF REPEATED ELECTROLYSIS.

The same unit was made cathode in one percent sulphuric acid solution under the same conditions for four successive runs without intervening rest periods.

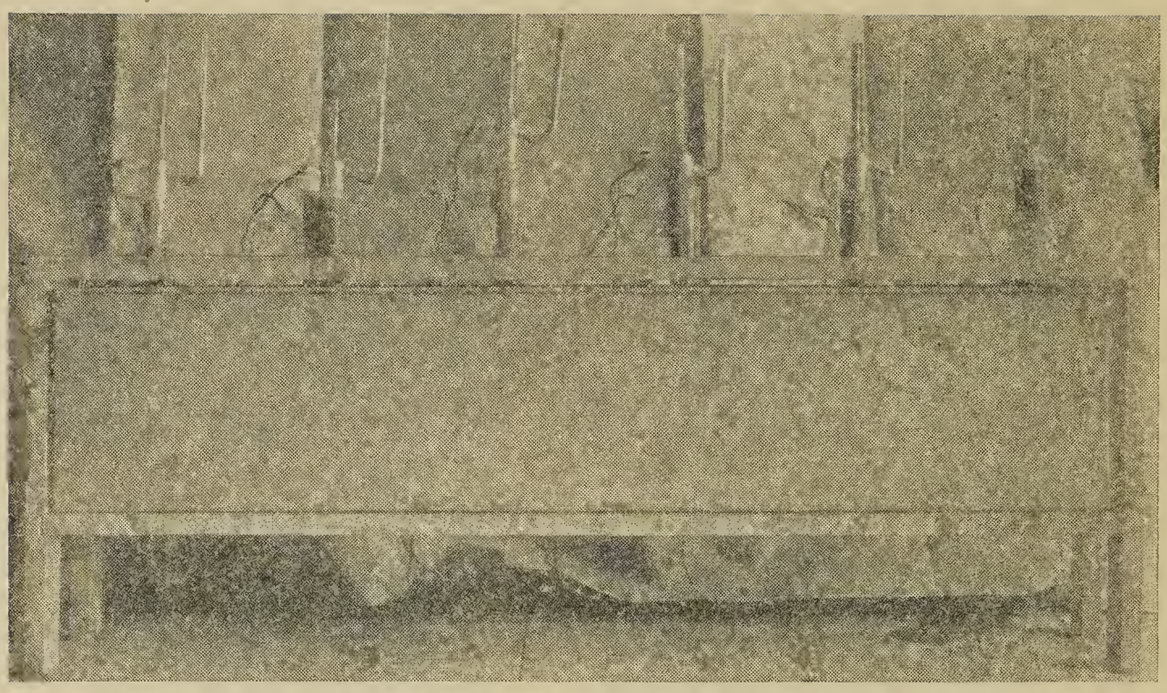

FIG. 4. Apparatus for Measuring the Rate of Hydrogen Penetration in Iron at Somewhat Elevated Temperatures, Showing Six Units Immersed in an Electrically Heated Water Bath.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron—New	0.5 Amp.	24 hrs.	20° C.	0.40 cc.
	"	39 "	"	1.35 "
	"	42 "	"	1.50 "
Same Unit as used above	"	24 "	"	0.30 "
	"	33 "	"	1.47 "
	"	48 "	"	2.00 "
Same unit as used above	"	5.75 hrs.	"	0.38 cc.
	"	6.25 "	"	0.46 "
	"	21.5 "	"	1.82 "
Same unit as used above	"	20 min.	"	0.08 "
	"	7.5 hrs.	"	2.10 "

The velocity of penetration increased with each successive electrolysis.

EFFECT OF ACID "PICKLE."

A unit which had been pickled for 48 hours in one percent sulphuric acid was immediately made cathode in the usual manner. The electrolyte was one percent sulphuric acid.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Pickled 48 hrs. in 1 percent H_2SO_4	0.2 Amp.	5.25 hrs.	20° C.	2.20 cc.
Same unit as used above	"	20 min.	"	0.20 "
	"	7.5 hrs.	"	2.10 "

The acid "pickle" facilitated the passage of the hydrogen enormously. The same volume of gas which penetrated this unit in 5 to 7 hours penetrated a new unit in 48 hours under the same conditions.

EFFECT OF REST OR HEATING.

Rest. A unit which had been electrolyzed five times, and whose penetration value was high, was allowed to rest for 72 hours and then made cathode in one percent sulphuric acid.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron — Previously electrolyzed five times, followed by a rest of 72 hours.	0.5 Amp.	23 hrs.	20° C.	0.25 cc.
	"	28.5 "	"	0.60 "
	"	31.5 "	"	0.92 "
	"	48 "	"	2.20 "

Heating. Another unit which had been electrolyzed a like number of times, and whose penetration velocity was therefore high, was heated to 130° C. for four hours in air and made cathode in one percent sulphuric acid.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron — Previously electrolyzed five times, followed by heating in air to 130° C. for 4 hours.	0.2 Amp.	24.5 hrs.	20° C.	0.03 cc.
	"	47.5 "	"	0.50 "
	"	60 "	"	1.28 "
	"	67 "	"	1.90 "

Both of these units, after their respective treatments, acted like new ones, that is, they showed penetration velocities equal to those of new tubes. The velocity of the latter unit which was

electrolyzed with a current of 0.2 amp. was less than the former whose current was 0.5 amp.

If units which have been operating as cathode continuously for several days, and which show a high penetration value, or if units which have been pickled for several days in acid be heated to a temperature of 130° C. for four hours or be allowed to rest for 72 hours, then they will behave like new ones, that is, their hydrogen penetration value will have been reduced to that of new units.

EFFECT OF TEMPERATURE.

A rise in temperature increased the penetration velocity very greatly. A unit was made cathode in one percent sulphuric acid at a temperature of 90° C.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron — Had been electrolyzed once followed by a rest of 72 hours.	0.2 Amp.	2 hrs.	90° C.	0.20 cc.
	"	2 " 50 min.	"	0.98 "
	"	3 " 20 min.	"	1.78 "
	"	3 " 30 min.	"	2.10 "

The volume of hydrogen which collected in this unit in 3½ hours was equal to the amount collected in a similar tube electrolyzed under similar conditions at room temperature in 48 hours.

Two more units were electrolyzed simultaneously under similar conditions at a temperature of 80° C. The two checked nicely with each other, but the rate of penetration was somewhat slower than the rate of the unit which was electrolyzed at 90° C. The slower penetration was probably due to the difference in temperature.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Previously electrolyzed heated to 130° C. for four hours.	0.2 Amp.	2 hrs.	80° C.	0.10 cc.
	"	3.5 "	"	0.80 "
	"	4.5 "	"	1.69 "
	"	4.75 "	"	2.00 "
Previously electrolyzed heated to 130° C. for four hours.	"	2 "	"	0.10 "
	"	3.5 "	"	0.60 "
	"	4.5 "	"	1.65 "
	"	4.75 "	"	2.00 "

The time required to collect 2 cc. of hydrogen at different temperatures, other conditions being equal, follows:

Temperature	Time
20° C.	48 hrs.
80° C.	4.75 "
90° C.	3.5 "

EFFECT OF VARIOUS ELECTROLYTES.

The rate of penetration varies with different electrolytes. Units were electrolyzed in one percent solutions of sulphuric acid, potassium sulphate, potassium hydroxide and tap water. The electrolysis in potassium sulphate was done at a temperature of 20° C. and is therefore compared to the behavior of a unit in sulphuric acid at that temperature, while the electrolyses in potassium hydroxide and tap water were done at 85° C. and consequently are compared to the behavior of sulphuric acid at that temperature.

Electrolyte: One Percent Potassium Sulphate.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron—New	0.2 Amp.	168 hrs.	20° C.	0.20 cc.
	"	216 "	"	0.25 "
	"	264 "	"	0.45 "
	"	312 "	"	0.58 "

Electrolyte: One Percent Sulphuric Acid.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron—New	0.2 Amp.	24 hrs.	20° C.	0.15 cc.
	"	39 "	"	0.90 "
	"	42 "	"	1.00 "

The rate of penetration for the unit immersed in potassium sulphate is very slow, being 0.0019 cc. per hour. The rate for the unit in one percent sulphuric acid is 0.024 cc. per hour, or at this

temperature twelve and one-half times the rate of the potassium sulphate electrolysis.

Electrolyte: One Percent Sodium Hydroxide.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron—New	0.2 Amp.	31.5 hrs.	85° C.	0.11 cc.
	"	40 "	"	0.37 "
	"	49 "	"	0.41 "
	"	60.5 "	"	2.00 "

Electrolyte: Tap Water.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Iron—New	0.2 Amp.	21.0 hrs.	85° C.	0.05 cc.
	"	30.5 "	"	0.85 "
	"	43.0 "	"	1.51 "
	"	61.5 "	"	2.00 "

Electrolyte: One Percent Sulphuric Acid.

Under the heading "The Effect of Temperature" it was pointed out that a unit electrolyzed as cathode in one percent sulphuric acid with a current of 0.2 amp. required 4.75 hours at a temperature of 80° C. and 3.5 hours at a temperature of 90° C. to collect 2 cc. of hydrogen. If it be assumed that the mean time or 4.125 hours would be required to collect 2 cc. of hydrogen at the mean temperature or 85° C. the following relation will hold:

Electrolyte	Time required to collect 2 cc. of gas under the same conditions at 85° C.	Relative Rate of Penetration
1% Sodium Hydroxide	60.5 hrs.	1.02
Tap Water	61.5 "	1
1% Sulphuric Acid	4.125 "	15

The time necessary to collect 2 cc. of hydrogen from an electrolyte of one percent sodium hydroxide or tap water, other conditions being the same, is 15 times the time required to collect 2 cc. from an electrolyte of one percent sulphuric acid.

HOT WATER AND STEAM.

Hydrogen produced by the reaction between tap water and iron, or steam and iron collected in the iron units at temperatures ranging from 50° C. to the boiling point. Tubes without electrical connections were immersed in water at 50° C., 90° C., and in steam.

Water at 50° C.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—New	3 hrs.	50° C.	0.10 cc.
	24 "	"	0.19 "
	84 "	"	0.22 "
	112 "	"	0.40 "
	172 "	"	0.51 "
	180 "	"	0.55 "
	188 "	"	0.60 "

Water at 90° C.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—New	24 hrs.	90° C.	0.30 cc.
	40 "	"	0.50 "
	48 "	"	0.69 "
	88 "	"	1.50 "

Steam at Atmospheric Pressure.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—New	2 hrs.	100° C.	0.15 cc.
	3 " 10 min.	"	0.20 "
	4 " 20 "	"	0.29 "
	5 " 25 "	"	0.38 "
	6 "	"	0.40 "
	7 " 15 "	"	0.50 "
	7 " 50 "	"	0.52 "
	9 " 10 "	"	0.57 "
	10 " 25 "	"	0.60 "

A straight line results from plotting the time required to collect equal volumes of hydrogen from the water-steam system, against the corresponding temperatures, measured on the centigrade scale.

The velocity of penetration of the hydrogen resulting from the reaction between steam and iron is greater at steam temperature than the penetration of the hydrogen of units made cathode in the usual manner in one percent sulphuric acid at room temperature, and less than the hydrogen of units electrolyzed, in one percent sulphuric acid at 90° C.

The relative rates follow:

Unit electrolyzed in 1 percent sulphuric acid at 90° C.....14
Unit immersed in steam.....2.5
Unit electrolyzed in 1 percent sulphuric acid at 20° C..... 1

PENETRATION OF COPPER AND NICKEL STEEL UNITS.

Copper Unit. A unit such as is shown in Fig. 1, was made up, having the tube *A* of copper instead of iron. After running as cathode under the usual conditions, namely, one percent sulphuric acid, 0.2 amp. and 20° C. there was no evidence of gas having collected at the end of 384 hours. At this point mercury had amalgamated with the copper to such an extent that the electrolysis had to be discontinued.

Nickel Steel Unit. Another unit was made up having the tube *A* of 3 percent nickel steel. This was electrolyzed under the usual conditions.

Description of Unit	Current	Time	Temp.	Volume of Hydrogen Collected
Nickel Steel	0.2 Amp.	4 hrs.	20° C.	0.04 cc.
	"	23.5 "	"	1.00 "
	"	29.5 "	"	1.20 "
	"	54.5 "	"	1.80 "

The penetration rate was roughly the same as the rate for iron under similar conditions.

PENETRATION OF IRON UNITS COATED IN DIFFERENT WAYS.

Four iron units, having the tube *A* of each unit treated with a different coating, were immersed in steam at atmospheric pressure. The coatings were tin (dipped), zinc (galvanized), zinc (sherardized), and copper (dipped).

Tinned Iron.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—Dipped in molten tin..	3.0 hrs.	100° C.	0.40 cc.
	8.5 "	"	1.12 "
	16.0 "	"	1.62 "
	22.5 "	"	2.00 "

Galvanized Iron.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—Dipped in molten zinc.	3.0 hrs.	100° C.	0.20 cc.
	20.0 "	"	0.72 "
	31.5 "	"	0.90 "
	45.5 "	"	1.10 "

Sherardized Iron.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—Heated in Zinc Powder.	1.5 hrs.	100° C.	0.15 cc.
	13.0 "	"	0.40 "
	28.0 "	"	0.45 "
	45.0 "	"	0.60 "

Coppered Iron.

Description of Unit	Time	Temp.	Volume of Hydrogen Collected
Iron—Dipped in molten copper.	40 hrs.	100° C.	.0 cc.
	44 "	"	0.10 "
	47 "	"	0.14 "
	103 "	"	0.72 "

In this experiment the hydrogen from steam penetrated the tinned iron unit much more rapidly than it does a unit of iron. The passage of the hydrogen was evidently facilitated by the presence of the tin. The rate for the two zinc-coated units was less than for iron. The rate for the coppered unit was very slow, and probably no hydrogen would have penetrated had the coating

been thick and uniform. It is likely that the hydrogen went through the iron and not the copper, in small areas where the latter had corroded away.

The comparative rates for iron, and for iron with the various coatings in steam, at atmospheric pressure follow:

Description of Unit	Time Required to Collect 0.60 cc. of Gas	Relative Rate of Penetration
Tinned Iron	4.0 hrs.	21
Iron	10.5 "	8.2
Galvanized Iron	15.5 "	5.5
Sherardized Iron	45.0 "	1.9
Coppered Iron	86.0 "	1

EXPERIMENTS WITH BARIUM CHLORIDE AND POTASSIUM DICHROMATE.

Evidence that the hydrogen which collected in the units was produced outside the tube and forced through the metal under pressure, and was not produced by the acid leaking into the tube, and later reacting with the metal, was furnished by two experiments.

(1) An iron unit was immersed, without electrical connections, in one percent sulphuric acid for 22 hours, during which time 2.4 cc. of hydrogen collected. The unit was then emptied of mercury and 10 cc. of distilled water poured in and shaken so as to come well in contact with the inside surface of the iron tube. This water solution gave a negative test for sulphates with $BaCl_2$, indicating that no sulphuric acid had come in contact with the inside of the tube.

(2) Another unit was immersed, without electrical connections, in a solution of one percent sulphuric acid to which had been added one percent of $K_2Cr_2O_7$. No gas collected in this unit after immersion for 96 hours.

The fact that the oxidizing action of the one percent $K_2Cr_2O_7$ in one percent sulphuric acid solution prevents the passage of hydrogen through the iron, at least for 96 hours, and that the inside of an iron unit pickled for 22 hours in one percent sulphuric acid showed a negative test for sulphates, furnish additional evi-

dence that the hydrogen which collects inside the units is formed on the outside and forced through the iron walls, and is not formed on the inside by reaction between the iron and some sulphuric acid, which has by some seemingly impossible means leaked through to the inside of the tube.

COMPOSITION OF THE GAS.

A sample of gas, which was collected in an iron unit electrolyzed as cathode in the usual manner, showed the following analysis:

Oxygen	None
Carbon Monoxide	"
Carbon Dioxide	"
Hydrocarbons	"
Hydrogen Sulphide	"
Hydrogen	94.5%
	94.8%

The remaining 5 percent was an incombustible gas such as N_2.

Another sample of gas, which was collected in an iron unit immersed in steam at atmospheric pressure, consisted largely of hydrogen.

CONCLUSION.

It has been shown that hydrogen penetrates iron at temperatures between 20° C. and 100° C. under a great variety of conditions, all of which influence the rate. The velocity of hydrogen penetration is greater for a unit immersed, without electrical connections, in one percent sulphuric acid than for units electrolyzed as cathode in a like solution, with such current densities as were tried. The rate for electrolyzed units is influenced by the current—the higher the current, for such densities as were used, the higher the rate—but the relation is not a straight line function.

The penetration velocity increases with each successive electrolysis, provided rest periods do not intervene, or with acid "pickling." The effect of rest or moderate heating upon units which have been electrolyzed or "pickled" is to restore the original resistance of the iron to the passage of hydrogen. Temperature has a marked effect, the rate of penetration increasing with the temperature. The rate at 90° C. for an iron unit made cathode in one percent sulphuric acid with a current of 0.2 amp. is 14

times its rate under similar conditions at 20° C. The velocities of penetration for units electrolyzed in one percent solutions of potassium sulphate and sodium hydroxide, and in tap water are about equal and are 1/12 to 1/15 the velocity of units electrolyzed in one percent sulphuric acid.

Hydrogen produced by the reaction between tap water at temperatures from 50° C. to 100° C. and iron, or between steam and iron, penetrates the metal at a rate depending directly upon the temperature. The oxide which is one of the products of the reaction forms a coating on the metal, which becomes thicker and thicker, and finally protects the iron from further action. If it were not for this coating of iron oxide, iron pipes carrying hot water or steam would continually give off hydrogen and quickly deteriorate.

The velocity of penetration for 3 percent nickel steel is the same as that for iron. Hydrogen does not penetrate copper at a temperature of 20° C. The rate for tinned iron is greater than for iron; and for galvanized, sherardized and coppered iron is less. It seems that the passage of hydrogen is facilitated by the presence of tin and retarded by zinc and copper. No evidence of sulphates could be found inside a unit which had been pickled in sulphuric acid and in which 2.4 cc. of hydrogen had collected. No hydrogen penetrated a unit immersed in a solution of one percent sulphuric acid plus one percent potassium bichromate in 96 hours. The gas which was collected in one of the units was analyzed and found to contain 95 percent hydrogen and 5 percent of incombustible gas, possibly nitrogen.

The facts will admit of the following explanation of the manner in which hydrogen is forced through iron tubes having walls 1/16-inch thickness. Atomic hydrogen (H) which has been liberated by the current, in the case of units which were electrolyzed, or by the reaction between metal and solution in the case of units which were not electrolyzed, penetrates the iron, where gaseous or molecular hydrogen (H_2) is later formed. Iron at room temperatures is impermeable to the latter. The atomic hydrogen continues to penetrate the surface of the metal rapidly and to form molecular hydrogen. The latter can escape only very slowly and as a pressure, sufficient to force the gas through the metal, is built up. It is a pressure built up in this way which also

results in the well-known phenomenon of the cracking of hardened steel when "pickled" in acid.

The writer hopes that the experiments which have been described in this paper will help to focus the thought of electrochemists on these problems and that as the result a more comprehensive understanding of the mechanism of the passage of hydrogen through iron at temperatures equal to or below the boiling-point of water may be gained.

Research Laboratory,
General Electric Co.,
Schenectady, N. Y.

DISCUSSION.

R. E. ZIMMERMAN[1]: Some three or four years ago we had approached this problem from a somewhat different standpoint, but I believe that some data were secured which might serve as a guide in further investigations. It might be of interest in connection with Mr. Fuller's paper to describe briefly the method we used and to mention some of the results obtained in this investigation. Our apparatus provided an annular space, one wall of which was formed by the piece of steel under investigation, the others being made of acid-proof material, and through the wall opposite the steel sample, connection was effected to a manometer or pressure tube. During a determination the apparatus was immersed in a glass vessel containing the acid solution, which in turn was placed in a thermostat to maintain the system at a constant temperature. By employing different acid concentrations and temperatures, we found certain characteristic periods which were called "penetration periods" or the time-intervals before penetration was effected in the case of different kinds of steel. Some of the points of interest brought out during the investigation were, first, that the time of penetration or the period before the pressure in the mercury manometer began to build up due to the passage of hydrogen through the steel wall, was practically independent of the thickness of the sheet used, which in this case was ordinary sheet steel from 30-gauge (0.3 mm.) to

[1] Director of Research Laboratory, American Sheet & Tin Plate Co., Pittsburgh, Pa.

as thick as 24-gauge (0.6 mm.). The carbon content and the chemical characteristics of the steel seemed to have a marked effect, the trend of the results being to show that the higher the carbon content the more rapid was the penetration. The physical condition of the steel was very important; that is, the extent to which it had been annealed or heat-treated subsequent to the rolling process. In general, unannealed steel was penetrated much more rapidly than annealed steel. We found, in agreement with Mr. Fuller's investigation, that temperature had a very marked and characteristic effect. The time-temperature function is not a straight line, for the first part of the range of increasing temperature caused a much more rapid penetration than that above 150° or 160° F. (66° to 71° C.). Mr. Fuller has taken considerable care to prove that the building up of pressure was not due to acid which leaked through the apparatus and then attacked the steel from the inside. I can say that we went through a similar period in the investigation and proved that the hydrogen actually did penetrate the steel. In some subsequent work we not only studied the passage of hydrogen through the steel, but the conditions under which some of the element is retained by the steel in such manner that it can later be driven out by heat. We hope that we shall be able to publish some of these results at a later date.

W. D. Bancroft[2]: This hypothesis, that what penetrates the iron is atomic hydrogen, is very interesting in its possible bearing on electrolytic reduction. It seems probable that the hydrogen over-voltage is due to atomic hydrogen, and that the metals which show the high over-voltage are the metals which do not catalyze rapidly the reaction from monatomic hydrogen to molecular hydrogen. From these experiments it appears that monatomic hydrogen penetrates iron readily and does not penetrate copper or zinc readily; and yet all those three metals have pretty high over-voltage. Consequently, it ought to be possible to work out some differences in electrolytic reduction due to the fact that the monatomic hydrogen does penetrate the iron in one case and does not penetrate the copper or the zinc in another case. It opens up a number of interesting possibilities and makes it seem probable that there are differences between copper, zinc and iron as

[2] Professor of Physical Chemistry, Cornell University.

cathodes which we have overlooked completely or have not been able to account for because we did not know these additional properties of monatomic hydrogen.

S. C. LANGDON[3]: We have been doing some work since the first of the year at the Bureau of Standards on the brittleness produced by pickling and plating, and we have obtained experimental data which, in many respects, confirm the data given in Mr. Fuller's paper, with reference to the rapidity with which brittleness effects are produced in steel, and which effects are assumed to be due to hydrogen. Taking a rod 0.37 inch (0.95 cm.) in diameter, the ability of that rod to withstand fatigue stress, that is alternating stress, is reduced anywhere from 30 to 50 percent by 5 minutes' immersion in 10 percent (twice normal) sulphuric acid at 50° C. We investigated different sizes of steel rods and rods with different carbon contents. The greatest brittleness was produced in the high-carbon steel that had been heat-treated and tempered; this same steel, which had been normalized, was less affected; that is, the brittleness was greatest for high-carbon heat-treated steel and decreased as the carbon content decreased; for steel which had been normalized, with the increasing carbon content the brittleness effect decreased. We arrived at the conclusion that the brittleness effect in steel was due to two factors, aside from any cleaning effect due to removal of grease; first, a temporary effect which was assumed to be due to hydrogen and which could be removed by an after-treatment; and a permanent effect, which was of much smaller magnitude and which we assumed to be due to the roughening of the surface. We worked with two types of material, rod and strip steel. We could get the same sort of effect by roughening the surface mechanically that we got by the etching effect of the acid. After treatment by heating in an oven or letting stand, the brittleness disappeared, that is, the temporary brittleness which we assumed to be due to hydrogen, would be gone in a week; it would be gone in two hours in an oven at 100° C., and would be gone in two minutes at 150° to 200°. We struck one thing there that did not correlate very well with this paper, and that was the fact that immersing this pickled steel in boiling water removed the temporary brittleness in from 2 to 10 minutes.

[3] Chemist, Evanston, Ill.

S. A. TUCKER[4]: In this connection it seems to me that the higher temperatures are concerned also in the fixation of nitrogen on the Haber plan. The Germans have taken out several patents on the chemical composition of the containers with a view to preventing this permeability of the container to hydrogen. I do not now recollect what analyses these give, but they have evidently taken this into account in patents now controlled by the Chemical Foundation. Evidently the chemical composition of the containers plays an important part at higher temperatures, around 500° or 600° C.

COLIN G. FINK[5]: In view of Mr. Fuller's findings as to the structure of hydrogen in steel, are we safe to assume that hydrogen in electrolytic iron is atomic hydrogen? We know that electrolytic iron is very brittle on account of the presence of hydrogen.

T. S. FULLER: The whole subject of iron and hydrogen is very complicated. It is true that the fatigue value of steel is cut down enormously by exposure to atomic hydrogen. The tensile strength, however, is not changed unless the steel be in a strained condition. If, however, we have a thing like a steel spring which is already strained, its tensile strength is cut down enormously. I think that the fact that heating a high-carbon steel which has been heat-treated and pickled in boiling water restores its fatigue value, is not inconsistent with this paper. The heating drives out the hydrogen and restores the fatigue value exactly as you do in heating an oven to that temperature, and the hydrogen reaction of iron in the hot water is so slow that there certainly would be no effect there in ten minutes; that is not inconsistent with the results of this paper. Dr. Fink asks if we are safe in assuming that the hydrogen in electrolytic iron is atomic hydrogen? I do not think so, for this reason: we made some experiments, first with a solution of potassium nitrate, made alkaline with sodium hydroxide. In that solution we dropped a piece of aluminum. Aluminum reacts with the alkali and produces nascent hydrogen, and the solution is thereby reduced to nitrite and ammonia. The odor of ammonia from such a solution may be easily detected. Some of the alkaline-nitrate solution was placed in a unit and electrolyzed for 24 hours, during which time much hydrogen came

[4] Chemical Foundation, Inc., New York City.
[5] Head of Laboratories, Chile Exploration Co., New York City.

off the inside of the unit and bubbled through the solution. The latter showed no tests for nitrites or ammonia at the end of the test. I think this is pretty good evidence that the hydrogen which comes off on the inside does so as molecular hydrogen. However, it certainly must go in as atomic hydrogen; and that is my reason for believing that pressure is actually built up there; I can see no other way in which the hydrogen can get through the metal.

W. D. BANCROFT: It seems to me that it ought to follow from this, that if you took a very thin sheet of iron and ran a heavy current through it, that you ought to be able to get some atomic hydrogen through.

T. S. FULLER: The sheet of the unit in which we placed this solution was 0.0015 inch (0.04 mm.) thick. I cannot agree with Mr. Zimmerman that the time of the first appearance of hydrogen on the inside is independent of the thickness of the sheet. The time for the first appearance of hydrogen on the inside of a unit one-sixteenth of an inch (1.5 mm.) in thickness is something over 15 hours; it appears within half an hour on the inside of this unit made of 0.0015 in. (0.04 mm.) iron.

R. E. ZIMMERMAN: I did not wish to be understood as making the statement quite as broadly or unqualified as that, that the penetration period was entirely independent of the thickness of the steel, because differences will certainly be found in extreme cases. However, there are so many other factors which cannot be kept constant, such as the physical condition and chemical characteristics of the steel, that they overbalance the effect of thickness alone on the penetration period. We had samples in which initial penetration was effected in 6 to 8 minutes, and the pressure of the mercury manometer went up steadily for probably 45 or 50 minutes until the steel was attacked so much that conditions were changed and no one knew what the results meant after that time. It was not uncommon to get pressures of 14 or 15 centimeters in the mercury manometer. I can say in confirmation of something that has been said here on the subject, that boiling the pickled steel in water did expel the hydrogen. There we are talking about the hydrogen that was occluded by the steel, not that which had passed through, but I do not quite understand

how you cure hydrogen brittleness simply by allowing the pickled piece to stand, for we had some steels which were so pickled as to occlude hydrogen, and which, after a week's standing, retained practically all of it.

F. C. FRARY[6]: With regard to the reduction of the nitric acid or nitrate to ammonia, I call attention to one possible error. I recall that the only cathode upon which nitric acid or a nitrate can be reduced to any extent to ammonia in an aqueous solution is the copper cathode. I am simply pointing out that there is a difference between the two conditions; in the sheet aluminum the gentleman had copper present; he had really an electrolytic cell: if he had an aluminum anode and a copper cathode, he would get the effect of the copper, which he might not get in the case of the iron surface. That will have to be tested before you can prove that you did not have the same form of hydrogen in both cases.

T. S. FULLER: The iron was Armco Iron.

WM. BLUM[7]: I would like to call attention to a practical application of this work of Mr. Fuller, and also of the experiments to which Dr. Langdon referred. Whatever the actual rate of penetration of the steel by hydrogen, the embrittling effect takes place so rapidly that for all practical purposes, in the short period used for pickling, a rod 0.37 inch (0.95 cm.) in diameter is affected just as badly as a rod 0.19 inch (0.48 cm.) in diameter. That is important because the first assumption, I think, is that such effects would be very much more marked on thin sheet material or small rods than on thick sheets or large rods; but the experiments show that in the ordinary periods of pickling, the embrittling effects on large rods are just as significant as on small rods. Another point, which it is hardly necessary to emphasize here, is that these effects are almost certainly due to the adsorption of hydrogen. There is a notion, more or less prevalent popularly, that you can eliminate the embrittling effect of pickling by subsequent treatment with alkali. If such effects are due to hydrogen, the alkali treatment could have no influence except insofar as the alkali happens to be hot, and therefore has the same effect as hot water. I mention this because within the

[6] Director of Research, Aluminum Co. of America, New Kensington, Pa.
[7] Chemist, Bureau of Standards, Washington, D. C.

last week I saw a specification in which it was stated that after pickling the work must be treated with alkali in order to remove brittleness.

COLIN G. FINK: I do not agree with Mr. Fuller in the conclusions he draws from his experiment in which he used the hydrogen escaping from his iron sample to reduce nitrogen. The hydrogen escaping from the iron may have been atomic to begin with, but changed to molecular at the surface of the iron. The experiment does not prove to my mind that the hydrogen present in electrolytic iron is either atomic or is molecular; I still hold that it may be atomic.

T. S. FULLER: I would like to ask Dr. Fink to explain the difference between the hydrogen escaping from the aluminum in the one case and reducing the nitrate and the hydrogen escaping from the iron in the other case.

COLIN G. FINK: In the case of the iron, we have an iron surface which readily catalyzes the atomic hydrogen to molecular hydrogen; but in the case of the aluminum, the reaction does not take place, the aluminum is not a good catalyzer for the reaction of atomic hydrogen to molecular hydrogen.

T. S. FULLER: I might also say that the hydrogen which comes through one of the units will not hydrogenate oil; I do not know whether that is of interest to you or not.

COLIN G. FINK: That does not help us.

S. C. LANGDON: Perhaps on one point I did not make as clear a contrast as I wanted to make, and that was in reference to the time required to remove the brittleness by heating in an oven at 100° for two hours; the same effect was obtained by heating two to ten minutes in boiling water. That was the question I was asking Mr. Fuller about. I was not saying that it was inconsistent at all with the paper; I was just curious as to his position on that point.

O. P. WATTS[8]: I would call attention to the use of the steel as anode in an alkali solution. I have found that in the case of watch-springs rendered brittle by hydrogen, the brittleness can be removed by using them as anode in a solution of sodium

[8] Associate Prof. of Chem. Engineering, University of Wisconsin.

hydroxide. In five minutes the spring is less brittle, and in half an hour the original elasticity is pretty thoroughly restored.

D. A. MacInnes[9] (*Communicated*): I differ from Mr. Fuller in his conclusion that "nascent" or atomic hydrogen (H) is responsible for the passage of hydrogen into iron during electrolysis or "pickling." In this connection I will take the liberty of calling attention to a recent paper on hydrogen over-voltage by the writer and Mr. Leon Adler.[10]

In the article mentioned, evidence is brought forward which leads to the conclusion that when hydrogen is evolved at the surface of a metal the greater portion of the hydrogen goes directly into the liquid, producing a layer of supersaturated dissolved hydrogen. The extent of this supersaturation depends upon the nature of the metal, the applied voltage, stirring, diffusion, etc. During the process of electrolysis there is a constant tendency for the liberated hydrogen to distribute itself between the three phases: solid, liquid and gas. Computation by the usual logarithmic formula indicates that even low over-voltages correspond to high concentrations of hydrogen—concentrations which would require very high external pressures to produce. The same article also includes a simple computation, based on Langmuir's experimental results, which indicates that the concentration of atomic hydrogen at low temperatures is very small indeed. The bearing of the above on Mr. Fuller's important investigation is this: During the evolution of hydrogen at the surface of iron, by electrolysis or "pickling," we have in all probability not H_1 or hydrogen at ordinary concentration, but a highly supersaturated solution of molecular hydrogen (H_2). This conception leads, I think, to an unforced explanation of nearly all of Mr. Fuller's results.

Donald P. Smith[11] (*Communicated*): These results are of much interest for the whole question of the mechanism of occlusion of hydrogen by metals, and the consequent diffusion. For they appear to show that the effect upon iron, of what may be termed a continued subjection to a pressure of atomic hydrogen, is opposite to that exerted upon palladium. With this latter

[9] Chemical Research Lab., Mass. Inst. of Technology.
[10] Journal of the American Chemical Society (1919), **41**, 194.
[11] Asst. Prof. Chem., Princeton University.

metal, Lessing[12] found that the rate of diffusion of electrolytically delivered hydrogen declined steadily during three successive experiments, before it finally became constant. Since both iron and palladium expand during occlusion, changes in the openness of their structures can hardly be supposed to explain an increase in diffusion velocity in the one case, and a decrease in the other. The inference seems, therefore, to be that alterations in the rate of diffusion, like those in electrical resistance and in certain other properties, are intimately connected with chemical combination, probably of the type usually designated as the formation of solid solutions. Such a chemical connection is of course not inconsistent with the existence within the interstices of the metal of both molecular and atomic hydrogen, for which there seems to me to be much evidence.

E. W. Kelly[13] (*Communicated*): Mr. Fuller states that copper is not penetrated by hydrogen. I have found under certain conditions that copper, nickel, silver and gold, besides iron, platinum and palladium, are readily penetrated by hydrogen gas. In the tests which I conducted I found it only necessary to heat these metals to approximately 400° C., when the hydrogen would penetrate at an appreciable rate. Palladium, as is well known, would permit the penetration of hydrogen at room temperatures, but this was greatly accelerated at 400°. The activity of the metals was in no sense uniform at the above-named temperature, but was accelerated very much by the increased temperature.

F. N. Speller[14] (*Communicated*): Mr. Fuller's experiments are of great interest, but exception should be taken to some of the conclusions expressed in the second paragraph of page 92.

The oxide (rust) formed by reaction between tap water or steam and iron is porous, and while this oxide probably slows down the solution of the iron and generation of hydrogen over a considerable surface, as Mr. Fuller points out, it tends to localize corrosion in the form of pitting, so that rather than affording final protection to the metal the metal is usually destroyed by perforation before 20 percent of the iron has been changed into oxide or rust. The dissolved oxygen in water must be taken into

[12] A. Lessing: Verh. d. deut. phys. Gesellschaft (1906), 4, 569-75.
[13] Victor Electric Corp., Chicago, Ill.
[14] Metallurgical Engr., National Tube Co., Pittsburgh, Pa.

consideration in this connection. We have found that corrosion is directly proportional to the free oxygen content of the water, temperature and other things being constant, so that it is fair to assume that the retardation in rate of hydrogen penetration was in part due to a lowering of the amount of free oxygen in the water as well as to the protecting action of the oxide formed by combination of this oxygen with the iron. In practice, experience indicates that only a thin film of oxide forms in a hot-water heating system (open at the top to atmosphere) and these pipes show no serious corrosion after 40 years' service. It is inconceivable that this film of oxide is responsible for the stoppage of corrosion. In fact, we now know by experiments that water free from dissolved oxygen is "de-activated" and practically inert to iron.

It would be interesting to know whether the author has made similar experiments in closed tanks, making determinations of the free oxygen present under various conditions and what the penetration of hydrogen is when free oxygen is absent or quite low. It seems to me further experiments are necessary to determine the relative effect of the amount of free oxygen present and the oxide film, in retarding the penetration of hydrogen.

Presented as part of a Symposium on "Catalysis" at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 26, 1919, H. S. Taylor in the Chair.

SOME PROBLEMS IN CONTACT CATALYSIS[1]

By Wilder D. Bancroft.[2]

There seems to be no doubt but that nickel splits ethyl alcohol into acetaldehyde and hydrogen because of the selective adsorption of hydrogen by nickel. The splitting of ethyl alcohol into ethylene and water by alumina is evidently due to the selective adsorption of water by alumina. In line with this is the fact that presence of water decreases the yield of ethylene in the case of alumina and that presence of hydrogen decreases the yield of acetaldehyde in the case of nickel. These results are entirely satisfactory qualitatively but they are not so good quantitatively. In the making of ethylene from alcohol for war purposes, it was found desirable to mix a lot of steam with the alcohol. One effect was a better heat control and the reaction was run at a higher temperature than would have been possible without the steam; but it is a little difficult to see how selective adsorption of water can play an important part in the presence of a considerable amount of water vapor. While the general theory is sound, there are details here which call for further study.

A catalytic agent ceases to function if it agglomerates so that its adsorbing power becomes negligible or if it adsorbs something which is not readily removed and which therefore cuts down the adsorption of the reacting substances. We know that if the reaction products are not removed rapidly from the catalytic agent, the reaction will slow down.

My attention has been drawn recently to several cases in which a reaction comes apparently to a standstill although no equilibrium has been reached. The experimental data are not as satisfactory as I should like; but I think that the results should be put on

[1] Manuscript received July 30, 1919.

[2] National Research Council, Washington, D. C.

record for what they are worth. Phosgene reacts with water to give carbonic dioxide and hydrochloric acid,

$$COCl_2 + H_2O = CO_2 + 2HCl.$$

So far as we know, this reaction is not reversible and it actually runs to an end in presence of an excess of water. In presence of concentrated hydrochloric acid the rate of hydrolysis is practically negligible. The only way that I can see to account for this is by assuming that water and phosgene do not react by themselves and that the reaction takes place solely in contact with the walls of the containing vessel. When these are coated with a film of hydrochloric acid of sufficient concentration, no phosgene is adsorbed to speak of and no reaction takes place. The hydrolysis should be studied with different concentrations of acid and with a varying ratio of wall surface to mass of solution.

Trichlormethylchlorformate, $ClCO_2CCl_3$, or superpalite as it has been called, decomposes to carbon tetrachloride and carbon dioxide in presence of alumina,

$$ClCO_2CCl_3 = CO_2 + CCl_4,$$

and to phosgene in presence of ferric oxide,

$$ClCO_2CCl_3 = 2COCl_2.$$

The reverse reaction has never been made to take place to any measurable extent. Some superpalite and ferric oxide were placed in a glass tube connected with a closed manometer. There was rapid decomposition at first, as shown by the increase in pressure; but, before long, the reaction came apparently to an end. On raising the temperature the reaction went a little farther and did not reverse when the temperature was brought back to its original value. This experiment was not checked sufficiently to make me willing to guarantee the results; but it looks as though the ferric oxide was poisoned and that when the temperature changed, more superpalite came in contact with the catalytic agent and was decomposed. If this is the true explanation, it suggests one interesting line of experimentation. When ethyl butyrate is treated with a small amount of enzyme, the decomposition only proceeds a little way. It seems probable that with an oscillating temperature it might be possible to carry the reaction much farther with the same amount of enzyme.

If we were to put calcium carbonate in dilute sulphuric acid and if the resulting calcium sulphate were to form a coherent film over the surface of the calcium carbonate, the reaction would come to an end as soon as the surface of the calcium carbonate was coated over completely. The apparent equilibrium would be more or less independent of the concentration of the acid. For a given size of calcium carbonate particles, apparent equilibrium would be reached when a definite amount of calcium sulphate had been reached. This is self-evident in a case of this sort; but the matter is not always quite so obvious. A certain war gas, which we will call *A*, gives a solid hydrolysis product. The same amounts of *A* were treated with 250 cc. of water and with 1,000 cc. of water. When apparent equilibrium was reached, the acid was four times as concentrated in the first case as in the second. Experiments were then made with dilute hydrochloric acid solutions instead of with water and it was found that the presence of hydrochloric acid in moderate amounts had practically no effect on the amount of hydrolysis. The hydrolysis took place in every case until the ratio of hydrolysis product to undecomposed substance was a constant. Of course it should have been shown that this ratio varied with varying crystal size; but there were a good many loose ends after the war and this was one.

Some recent experiments by Lind[3] can be looked upon as a displacement of equilibrium by a catalytic agent if one so wishes. Radium emanation decomposes liquid water into hydrogen and oxygen; but causes hydrogen and oxygen to combine. If the emanation is placed in the liquid phase, hydrogen and oxygen will be formed and will escape into the vapor phase. If the emanation is moved up into the vapor phase, the reverse reaction will take place.

Cornell University.

DISCUSSION.

H. S. TAYLOR[1]: The chemical engineer, when he wishes to make a reaction go, has generally employed, in the past, four agencies: he has used temperature, pressure, solubility, or the

[3] Trans. Am. Electrochem. Soc. (1918), 34, 211.

[1] Instructor in Physical Chemistry, Graduate College, Princeton, N. J.

electric current. The more modern the chemist is, the more he looks to a new agency for the solution of many of his problems. That agency he utilizes, along with temperature, pressure, solubility or the electric current. In order to effect his purposes, he uses a catalytic agent. It is this catalytic agent that we wish to discuss in some sort of detail this morning. We wish to get an idea, first of all, as to what a catalytic agent is. Mr. Hendricks, whom most of you know, calls the catalyst the human mischief-maker. The editor of a Vermont newspaper, attempting to tell his readers what Mr. Ellis is calling to your attention in the article he is distributing today, called the nickel catalyst "The Chemical Parson." I believe in that definition for nickel. In the catalytic hydrogenation of fat, nickel is "the chemical parson" since after reaction it is free to bring about the union of further amount of the reactants. The catalytic substance will remain unchanged upon completion of the reaction, unless, of course, secondary factors enter in, which would have to be discussed independently. Since the catalyst remains unchanged, it is able to bring about the combination of large quantities of the reacting substances. That is why, for example, a single platinum gauze placed in the path of a stream of ammonia-air mixture is sufficient for the oxidation of sufficient ammonia to supply oxides of nitrogen to a sulphuric acid plant making 60 tons a day. If you pass nitrogen and hydrogen through a tube at 600°, nothing will happen until you put a small amount of catalyst in the path of the nitrogen and hydrogen, when they manifest their normal chemical affinity and unite to form ammonia. That is the basis of the process which has been used during the past four years to supply a considerable part of the fixed nitrogen requirements of Germany. The hydrogenation of fats is a large industry of 10 or 15 years' growth. In that process the hydrogen is passed into a fat medium into which a suspension of nickel or some suitable catalytic agent, generally nickel, is placed. The hydrogen is adsorbed, and the unsaturated glyceride is converted into a saturated glyceride, a change from liquid to solid fats which have been shown to be edible, and suitable for the manufacture of soaps. Dealing with an agency which is capable of such wide application as this, it is obvious that we have a great deal to discuss. Mr. Hendricks' definition of the catalyst as "a human

mischief maker" arises largely because some of the operations of the catalytic agent are, as far as we know at present, quite mysterious, or at any rate they have been so regarded in the past. It is our object, as far as possible, to remove those mysteries from the sphere of science. Secondly, owing to the fact that certain substances will catalyze a reaction and other substances will have no effect whatever, we have a certain specificity on the part of the catalytic agent which is also a matter of difficulty and a matter for discussion. Then there are other kinds of problems which arise when it is attempted to transfer a known catalytic process from the laboratory scale to the technical scale. Consequently we wish to discuss this morning some of those aspects. First of all, Dr. Bancroft will point out to us some of the problems that still seem strange from the point of view of the ordinary investigator. I would like to add some more of those problems. Mr. Zeisberg and Dr. Huff are going to treat of the thermal problems in contact catalysis. When a reaction has been studied, when a suitable catalyst has been found, one generally finds that either so much heat is required or so much heat is given out in the process that it is a matter of difficulty to control the temperature of the catalyst, due to local overheating or local cooling. In respect to that problem we will fortunately obtain Mr. Zeisberg's views on the contact sulphuric acid process, and he has been able to give us a resumé of how the problem was handled in actual practice. Dr. Huff has given us a further contribution to this problem on the organic side. Finally Dr. Rideal, of the University of Illinois, is going to treat some of the problems of catalysis in relation to the modern theories of matter. The main object in getting together a symposium on catalysis was to elicit views from the audience with regard to the different aspects of catalysis; therefore it is hoped that a large part of the meeting will be held on the floor of the hall.

COLIN G. FINK[2]: Dr. Bancroft defined the poisons of a catalytic agent as such substances as would interfere with the adsorbing qualities of the catalytic agent. I wonder whether there is any reaction known where the reacting substances, say the reacting gases, are influenced, or affected as far as their adsorbing tendency is concerned? In other words, say, we had a gas, B,

[2] Head of Research Laboratory, Chile Exploration Co., New York City.

which had a tendency, a great tendency, to be adsorbed by platinum. Isn't it conceivable that by the introduction into your reaction vessel, of some other gas the adsorbing quality of the gas might be greatly reduced irrespective of the platinum catalyst? Couldn't we poison the reagent without poisoning the catalyst? I do not know of a case; I am just asking a question. I feel certain there must be cases such as these.

W. D. BANCROFT: I do not know; I should doubt it. Of course you get the other side, hydrogen and ethylene with palladium; we know that palladium takes up hydrogen tremendously. The presence of ethylene vapor on the surface checks the adsorption of hydrogen by palladium; it cuts it down from 900 volumes to practically nothing, but that is due to the adsorption of the gas on the palladium. I do not quite see how anything not adsorbed by the catalytic agent could affect the adsorption of one of the gases. It is perfectly free and untrammeled, by definition; it can do whatever it likes. If your hydrogen is unchanged and your catalytic agent is unchanged, I do not see any way in which the presence of another substance in the gas phase could cut down the adsorption, but maybe that could be done.

COLIN G. FINK: Let us reverse the case; say we have a gas which is not readily adsorbed; can we not conceive of activation taking place possibly through radium or something of that kind? The gas is more or less inert, at first, as far as adsorbing quality goes; then it will be activated and after that readily adsorbed. I do not think we ought to limit ourselves to Prof. Bancroft's definition, that a poison in a case of catalytic reaction is a substance which interferes with the adsorbing qualities of the catalytic reagent alone. It may interfere with the adsorbing properties of the reacting substances.

W. D. BANCROFT: There is one case, of course, where the thing could come in; it has just occurred to me. If, instead of a gas, you take a liquid, you can cut down the adsorption; for instance, charcoal adsorbs dyes from an aqueous solution, certain dyes, very readily. From alcoholic solutions, in which those dyes are much more soluble, the charcoal adsorbs much less, and consequently to that extent you would have a change in the amount of adsorption which is quite independent, or might be

quite independent, of any reaction between the other two things. So if you get up to gases under very high pressure, where you could consider the solvent action of the gas on the substance, it seems to me that the case cited by Mr. Fink could occur perfectly well. In fact we know it does occur with liquids. Whether it could occur under any ordinary pressures with a gas, I am very doubtful because we have no evidence of any solvent action of a gas at ordinary pressures.

M. L. WEISS: Dr. Bancroft mentioned a catalyst that had an affinity either for the reacting substances or for products of reacting substances. There has also been mentioned the use of platinum as a catalyzer in the hydrogenation of oils. Platinum is known to have the power of diffusing gases, and therefore has an affinity for some of the reacting substances. I have known a catalyst that had no affinity for any of the reacting substances and no affinity for products of the reacting substances. Take a case of organic chemistry, such as aniline dyes; when aniline is brought in contact with carbon bisulphide, thiocarbanilid is formed. It takes a long time before thiocarbanilid is formed, and the reaction is never complete, but when a catalyst is used, the reaction is brought to completion in a manner dependent upon the nature of the catalyst and on the time and temperature. When potash is used as a catalyst, it is understood that the catalytic action would be to take up the hydrogen sulphide as potassium sulphide. I have used a catalyst that has no affinity for hydrogen sulphide; used in small quantities, it has no adsorption affinities or properties and causes the reaction to reach completion in a comparatively short time. I cannot explain the action of that catalyst.

W. D. BANCROFT: Do you mind telling us what the catalyst is?

M. L. WEISS: I am not at liberty to tell, but if it is used in small quantities it has no affinity for calcium sulphide; the reaction will go on without a rising temperature, but it takes a longer time. The catalyst in this case has no affinity for any of the reacting substances or products of the reacting substances. I cannot very well explain the reaction of this catalyst. If Prof. Bancroft or any of the audience can enlighten me, I will be very glad.

W. D. BANCROFT: Is it the case of a solid catalyst?

M. L. WEISS: Yes.

W. D. BANCROFT: How do you know it does not adsorb some of these things?

M. L. WEISS: The hydrogen sulphide evolved has been measured in given quantity.

W. D. BANCROFT: You could not make the measurement accurately enough for that.

H. S. TAYLOR: As a matter of fact, that is the whole point at issue. Everybody would say that reduced iron had no affinity for water at all at a temperature of 500° C. As a matter of fact, if you pass nitrogen and hydrogen at 100 atmospheres pressure and containing 0.01 percent of water vapor over an iron catalyst at 500° C., the water vapor is largely retained by the iron catalyst and this causes a decrease in efficiency as an ammonia-making catalyst by at least 20 percent. Two days ago Dr. Chaney told us it was not possible to remove all the hydrocarbon from charcoal at a temperature of 900° C., which shows that the charcoal has a great affinity for hydrocarbon in small quantities, even at a high temperature. It is difficult to state off-hand that a substance has no affinity for a given catalyst; it may have an enormous effect, such as the presence of 0.01 percent of water vapor on iron.

W. R. MOTT[3]: The gentleman referred to the photochemical reaction of light on chlorine. It happened before the war that I did a little work on chlorine in the bromination of light, and ran across a number of uncertain results. I noticed in Reather's reaction for the production of methylchlorine that a minute amount of iron is necessary; it is the most delicate test for iron there is, and you have to have an adsorption coefficient in your gas that will take the light before the light will react. Often by adding a colored medium you can cause the light to make a catalytic change.

V. R. KOKATNUR[4]: I want to go back to the decomposition of superpalite and ask if the author can suggest why ferric oxide decomposes at this point and why alumina decomposes at this point? Would it not be possible, under these circumstances that

[3] National Carbon Co., Cleveland, Ohio.
[4] Research Chemist, Niagara Alkali Co., Niagara Falls, N. Y.

iron, which we know forms a compound with carbonate, might decompose it completely into iron carbonate and chlorine, and later carbonate and chlorine may form phosgene? I was going to ask if Dr. Bancroft could suggest why iron oxide decomposes superpalite at this point, and why alumina decomposes at this point? He suggested that the reaction is very similar in the two cases, but as a matter of fact you will see it is not so similar, there is a slight difference, and I want to know why iron oxide should decompose at this point and why alumina should decompose at this point. I wanted to ask if he could suggest anything on that?

W. D. BANCROFT: I have not the remotest idea off-hand, and I should expect them both to react the same way. I do not know whether they do react that way. I merely said that that was a possible explanation which would account for your getting different products although in both cases you were breaking the bond between the carbon and the oxygen.

Presented as part of a Symposium on "Catalysis" at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 26, 1919, H. S. Taylor in the Chair.

FURTHER PROBLEMS IN CONTACT CATALYSIS.[1]

By Hugh S. Taylor.[2]

In a series of communications,[3] Bancroft has recently collected a considerable body of evidence relative to the adsorption theory of contact catalysis and to the part played by selective adsorption in determining reactions of this type. They include, perhaps, the most interesting of the catalytic reactions since, owing to specificity as to catalytic agent and to reaction product, the most varied reactivities of chemical materials may be achieved. Where, demonstrably, intermediate compound formation occurs during the operation of the catalytic process, the interest is lessened, since in such case the catalytic reaction becomes simply the occurrence of several successive reactions. The velocity with which the reactions succeed each other in the catalytic process as contrasted with their rate of occurrence when separately conducted constitutes their main problem of interest. With respect to the catalytic actions in which intermediate compound formation or carrier action is not demonstrable it will be useful briefly to examine the features which they present and the adequacy of several viewpoints which have been advanced in explanation of the phenomenon.

The first striking feature common to most of the contact agents successfully employed in catalytic reactions is their structure. Contact agents are, in the main, porous or finely divided materials. This is well illustrated by the chief industrial catalysts. Bog iron ore, a light porous hydrated oxide of iron, is the agency for the fractional oxidation of hydrogen sulphide to sulphur in the purification of illuminating gas. Cuprous chloride distributed over a porous support, such as firebrick, is used in the Deacon chlorine

[1] Manuscript received September 15, 1919.

[2] Professor of Physical Chemistry, Princeton University.

[3] Journ. Physical Chem. (1917), 21, 573, 644, 734; (1918), 22, 22.

process. Platinum deposited on asbestos or on salts like magnesium sulphate is used in the contact sulphuric acid process. Finely divided nickel on porous supports is used for hydrogenation of fats. Porous reduced iron-molybdenum is a successful catalyst for ammonia synthesis; massive ferro-molybdenum is quite inactive. A platinum gauze, initially of little activity in the oxidation of ammonia acquires activity steadily with use and is simultaneously converted from a smooth plane-surfaced wire to a corrugated, pitted material as is well shown in the photo-micrographs recently published by Parsons.[4]

These porous materials are also characterized by their adsorbent properties. They adsorb greater or lesser quantities of gases so that, naturally, an association has been made of the two phenomena of catalysis and of adsorption. Nevertheless, the two phenomena are, in certain examples, sharply distinct. Thus Dr. W. A. Patrick, of Johns Hopkins University, has called my attention to the fact that his silica gel, whilst showing excellent adsorptive power, does not to his knowledge, catalyze any reaction, of many studied, other than that of the combination of nitric oxide and oxygen.* We must therefore conclude that all substances which adsorb a given set of possible reactants will not promote reaction between them. This point may be further illustrated in connection with the oxidation of carbon monoxide to carbon dioxide, whether this be conducted in presence of other combustible gases or in simple mixture with air. As is now well known, the oxidation reaction may readily be brought about in the presence of metallic oxides and more especially in the presence of mixed oxide catalysts, the activity of which is so great that reaction may proceed at ordinary temperatures and with readiness at 100° C. The simple catalytic oxidation at ordinary temperatures is being applied to the problem of removing carbon monoxide in contaminated air, as for example in mines, whilst the preferential oxidation process is being employed in the removal of carbon monoxide from industrial hydrogen, a process which was worked out in the research laboratories of the British Munitions Inventions Department. Now charcoal, especially the more reactive types developed recently, shows marked adsorptive action towards

[4] Journ. Ind. Eng. Chem. (1919), **11**, 550.

* It is interesting to learn that silica gel is catalytically active in several dehydration processes.

carbon monoxide, oxygen and carbon dioxide. Its catalytic activity in promoting the oxidation process on the other hand is, as contrasted with the metallic oxides, practically *nil*. Here then is a problem of relative simplicity which should yield to investigative work, unless a solution of the problem can be offered from available experimental material. Ethylene and oxygen react at ordinary temperatures in presence of charcoal to form carbon dioxide and water. Does the sharp differentiation between carbon monoxide and ethylene with this particular contact material lie in the quantitative data of the adsorption effect of charcoal on the two gases? And if so, does a similar differentiation in the quantitative data relative to adsorption of carbon monoxide, by charcoal on the one hand and by metallic oxides on the other hand account for the varying catalytic activity of these two sets of substances? It is unfortunate that a small amount of data collected in this country during the war is not yet availaible to help towards a decision in this relatively simple case, so that it becomes necessary to repeat some of this work. Not only is it necessary to know the relative amounts of each gas which may separately be adsorbed, but it is further necessary to know what relative amounts of the gases are adsorbed from a mixture of the gases. This latter is an important point in adsorption, data concerning which seem relatively scanty. The influence of one gas on the adsorption of the other is quite marked as is shown by the following figures of Dewar[5] for the adsorption by 1 cc. of charcoal at 0° C. of different gases measured in cubic centimeters at 0° C. and 760 mm. pressure.

Adsorbed gas	H_2	O_2	CO	$2H_2+O_2$	$2CO+O_2$
Volume adsorbed	4	18	21	12	30

Also, it is of importance to know how far the adsorption observed is reversible. The extraordinary nature of the adsorption of gases accompanying chemical change is well illustrated in the recent publication of Tideswell and Wheeler[6] in connection with the oxidation of coal. It is there shown that in presence of air and 7.45 percent CO, of an initial atmosphere containing, *inter alia,* 590 cc. of oxygen, as much 511 cc. were adsorbed at 100° C. in 120 hours. Simultaneously 52.5 cc. of CO_2 were

[5] Ann. chim. phys. (1904), (8), 3, 5.
[6] Journ. Chem. Soc. (1919), 116, 895.

formed and 22 cc. of CO. The gases removed from the coal by exhaustion at 200° C. consisted of 26.5 cc. of CO_2, 7.7 cc. of CO, 0.3 cc. of N_2 and *nil* of O_2 in spite of the large quantity of this latter gas which had disappeared in the operation. Using a hydrogen air mixture it was noted that oxygen disappeared as before, CO_2 and CO were similarly produced, the hydrogen remained unchanged, nor was it to be found in the gases obtained by evacuation of the coal at 200° C. An extension of this type of work to other catalytic agents would be of the highest interest.

The effect of one gas on the adsorption of a mixture of gases brings up the general problem of p o i s o n s in contact catalysis, in the discussion of which Bancroft has attempted "to show that we can account for all the cases that have been considered, if we postulate suitable and not improbable adsorptions. To make the case complete one ought to show that al l substances which poison catalytic agents do cut down the adsorption of the essential reacting substances or reaction product." It would seem, however, that the negative catalytic action exerted by small traces of the so-called poisons may possibly be accounted for in a somewhat different manner. Hitherto in applying the phenomena of adsorption to catalysis it has been customary to think in terms of the equilibrium amount adsorbed. Recent developments seem to indicate, however, that it would be well to consider also the time-factor or velocity of adsorption. Normally the time of contact between a gas and the solid catalyst is extremely brief, frequently of the order of a small fraction of a second. Now, while, admittedly, the velocity with which adsorption takes place is usually extremely rapid, it is obvious that in operations involving brief periods of contact this velocity of adsorption may be of the utmost importance. So we may venture to think that t h e e f f e c t o f c a t a l y s t p o i s o n s m a y b e d u e i n p a r t t o a r e d u c t i o n i n t h e v e l o c i t y w i t h w h i c h a d s o r p t i o n o f t h e r e a c t i n g s u b s t a n c e s o c c u r s. It is believed that this viewpoint with regard to the problem of catalyst poisons is essentially new and it opens up an interesting line of experimental work with a view to test of the same. Certain data in connection with charcoal adsorption seem undoubtedly to give credence to the viewpoint. Thus, recent ex-

perimenters have frequently emphasized the importance of using "clean" charcoals in the study of charcoal adsorption. The previous life history of a charcoal largely influences its adsorptive efficiency in the gas mask. And, of actual velocity measurements one at least is forthcoming in a communication made privately to the author by Dr. Harned, of the University of Pennsylvania.[7] He has obtained curves dealing with the velocity of adsorption of chlor-picrin using ordinary gas mask charcoal, (a) which has been cleaned by successive washings with chlor-picrin,

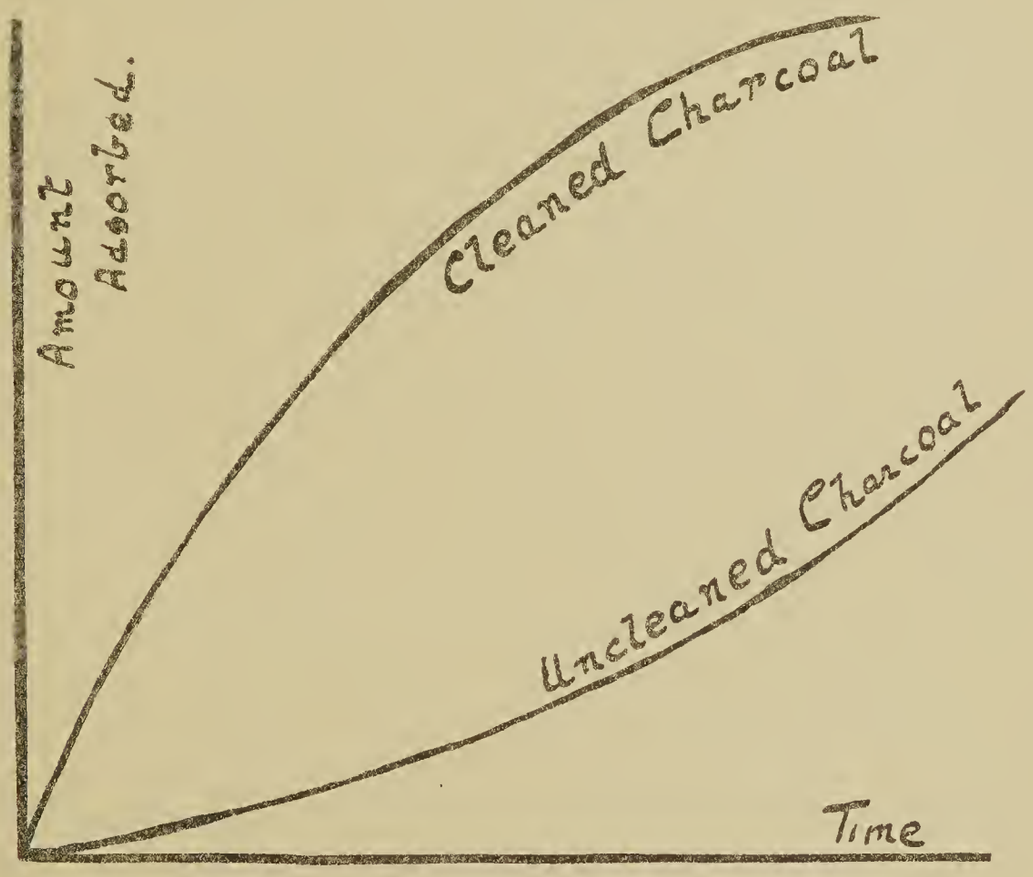

FIG. 1.

(b) which has been simply evacuated before use. Harned has shown that the run of the curves in the two cases is as shown in Fig. 1. It will be seen that in an indefinitely long interval of time the amount adsorbed tends to the same value in the two cases. With the clean charcoal, however, the velocity of adsorption is considerably greater in the earlier stages of the process than with the charcoal which has not been cleaned. Thus, in a given experiment, in the first 5-minute interval the velocity of ad-

[7] See also: McGavack and Patrick, Journ. Am. Chem. Soc. (1920), 42, 946, for the influence of air on the velocity of adsorption of sulphur dioxide by silica gel.

sorption was some 500 times greater with the cleaned than with the "uncleaned" charcoal. For a briefer interval such as is usual in catalytic actions the ratio of the two would be more markedly diverse than in the 5-minute interval, and could thus be of the same marked order of divergence as obtains with catalytic reaction velocities in presence and absence of traces of catalyst poisons. It is to be understood, however, that such considerations do not refer to those catalyst poisons which definitely change the chemical state of the catalyst or alter its physical state by deposition of layers of solid material or their equivalent. In these cases poisoning is progressive and permanent.

Bearing in mind the inefficiency of the silica gel as catalyst, it cannot be excluded from account that apart from the quantitative factors of adsorption involved the nature of the adsorbent or catalyst may play a large and determining part. What is happening in the activation of a platinum gauze during ammonia oxidation? It is evident from the physical state after activation that large energy changes have taken place at the surface. Chemically the material is the same as far as is demonstrable. That the chemical reactivity of the material for simple reactions, however, is vastly changed can be emphasized by record of a similar example. If hydrogen be passed over the copper oxide wire of commerce, rapid reduction begins in the neighborhood of 300° C. If the copper so formed be oxidized at a somewhat lower temperature it will be found that subsequent rapid reduction will occur at a lower temperature than in the first reduction. This process of oxidation can be repeated to such a degree that the wire becomes sufficiently reactive to be reduced or oxidized at 100° C.; and, examination of the wire shows that it is in a similar physical condition to that of the activated platinum. Such copper oxide, moreover, will c a t a l y z e rapidly the combination of hydrogen and oxygen at 100° C., whereas charcoal will be inert. We see therefore that in increasing the chemical reactivity of the contact agent we have increased its catalytic activity.

Now the results thus obtained by careful preparation of the catalyst in a highly reactive state with simultaneous variation of

its physical characteristics are frequently more readily produced by the use of promoters. Normally it would be anticipated that the catalytic activity of a mixture should be composite of the several activities of the components and such is of very frequent occurrence. Nevertheless exceptions are well known and it has often been shown that the admixture of small quantities of other substances with single catalysts results in abnormal increase in catalytic activity of the mixture. Thus, a mixture containing 2.5 percent Cr_2O_3 and 0.5 percent CeO_2 with iron oxide gives a catalytic agent for the interaction of steam and carbon monoxide many times more reactive than iron oxide alone unless this latter is submitted to exceedingly careful preparational treatment. To such admixtures the name of catalyst promoters has been given. A large literature on the subject of promoters now exists, which it is hoped shortly to publish from the writer's laboratory,[8] but which is beyond the scope of the present communication. As yet, however, the explanation of promoter action is not forthcoming. Indeed it is probable that no one single theory would be generally applicable. In certain cases, as for example, in the use of molybdenum as a promoter of iron in ammonia synthesis, the phenomenon may be associated with the nitride forming or nitrogen adsorbing properties of molybdenum. In cases of carrier action, as for example, in oxidation processes, where catalytic action may possibly be associated with the tendency of an element to oscillate between two stages of oxidation the function of the promoter may be to increase the temperature range over which the catalytic agent is labile. This point can readily be tested experimentally and is the object of some investigations at present in progress in the writer's laboratory. It would seem in other cases that promoters may act by decreasing the sensitivity of the catalyst to substances which diminish its activity. Thus, admixture of copper with nickel in the hydrogenation of oils appears to increase the ruggedness of the catalytic agent and to render it less sensitive to traces of such negative catalysts as carbon monoxide. In these several branches the problems ahead are numberless. The results from their investigation will be very valuable.

[8] See Pease and Taylor, Journ. Physical Chem. (1920), **24**, 241.

DISCUSSION.

S. C. LIND[1]: The great renewal of interest in the subject of catalysis that has been produced by the war is extremely interesting. Of course any knowledge of the subject of catalysis had to come after some knowledge of the subject of kinetics; that is, the rate at which chemical reaction takes place under ordinary circumstances, and I think we should not lose sight of the fact that catalysis is simply allowing something to take place that wants to take place anyhow. To my mind it is more surprising that we find as much regularity as we do in the kinetics of reactions than that the opposite should be true. My idea of catalysis has always been (and there is no originality in the idea at all: I am sure that many of you hold the same idea) that it is simply a term for a rather heterogeneous collection of phenomena and terms which gradually we will be able to classify and explain. The explanations for all the different kinds of catalysis may turn out to be the same or may turn out to be something quite different. This morning the discussion has been confined almost entirely to what we have known as contact catalysis. I hope that the discussion will not be entirely confined to this class, nor do I think we ought to lose sight of the fact that although poisons have been spoken of as inhibitive contact agents, we have cases of negative catalysts that act entirely homogeneously, where contact catalysis plays no rôle whatever. For example, in the reaction between hydrogen and bromine, which is a well-known reaction that takes place entirely in the gas phase, you can increase the surface of the reacting vessel several fold or any amount you wish without affecting the velocity of the reaction at all, and yet the hydrobromic acid gas being produced in the reaction has a very marked negative catalytic effect on the reaction. A much more striking case is the inhibitive effect of oxygen in the photo-chemical reaction between hydrogen and chlorine; an extremely small quantity of oxygen inhibits that reaction, and very interesting explanations have been recently proposed for that inhibitor. As long as we find these cases in pure homogeneous gas reactions, of a pronounced similarity, I do not see the necessity of any special ex-

[1] Chem. in Radioactivity, U. S. Bureau of Mines, Golden, Colo.

planations for those reactions in the case of contact catalysis. Dr. Taylor's theory is an extremely interesting one, and it may or may not turn out to be true. It is certainly worthy of very careful investigation, but to my mind such an effect in the case of contact catalysis may be simply due to the rather enhanced effect that we know to take place on the surface; in other words, it may be rather an extreme case of what may be already taking place in the gas phase.

Just how rapid will be the progress of removing catalytic reactions out of the general grab bag in which they have been placed and putting them on a truly scientific basis, is a little difficult to be seen. A few cases have been already so removed. I have been interested for several years in the other side of catalysis, which has been lightly touched on this morning, namely, the reactions brought about by the influence of some form of radiant energy. The most important case in nature is that of photo-chemical reactions. They are of extreme importance, and when you come to realize that all the coal we have stored up and practically all our sources of available energy except water power, have been produced by some form of photo-chemical reaction, you realize how important they are and how little we know about them. Yet I have not been directly interested in them so much as in the action of another form of radiant energy, some of the radio-active rays producing chemical reactions, and I am very glad to be able to tell you that this is one set of reactions which were regarded as catalytic which we can now remove entirely out of that class. They are not catalytic reactions any more than the passage of the electric current and the chemical actions accompanying it at the electrode, and are not catalytic because there are perfectly definite relations between the amount of electric or kinetic energy involved and the amount of reaction produced, which is not the case for the action of catalysts. Then there is one characteristic the reverse of that pertaining under ordinary conditions: the catalyst usually acts in solutions somewhat roughly in proportion to the concentration, whereas radio-active catalysts act in reverse order, the explanation of which is very easy. If we have a certain amount of radium emanation in a confined volume in any gaseous system, if we increase the volume without changing anything else, also leaving the gas pressure constant, the amount of

reaction is increased directly in proportion to the diameter of the vessel. That is due to the length of path of the Alpha rays, but you must remember that catalysis is universally proportional to the volume or to the cube of the diameter, so it is a reversed case to a certain extent, though easily explainable. So far as photo-chemical reactions go, a good deal of progress is being made in the direction of removing them from that general uncertain category. I would like to mention one interesting case in closing. It is possible that it has not come to your attention on account of the great and unfortunate disturbance in receiving German literature during the war. About two years ago Prof. Nernst proposed the most satisfactory explanation of the photo-chemical action between hydrogen and chlorine I have seen, and since that is one of our most typical photo-chemical reactions, I would like to give you his explanation in detail, because you may not have been able to see the original article. Prof. Nernst's explanation is purely thermo-dynamic; it does not involve any unusual assumptions further than the assumption of a primary dissociation of hydrogen and chlorine into atoms by light, which we know is quite possible from Einstein's photo-chemical law. This, of course, is the primary reaction that takes place. We have present only two gases to begin with; suppose we have a certain wave length acting which is capable of dissociating, let us say, chlorine into chlorine atoms (a very small number, of course, out of the total number of molecules present) then we can have the following reaction take place, forming what we used to speak of as an intermediate product in these reactions: One molecule of hydrogen combining with an atom of chlorine to form this unstable product which immediately breaks down further into ordinary HCl plus hydrogen atoms; $Cl + H_2 = H_2Cl$ and $H_2Cl = HCl + H$, According to Prof. Nernst's theory then, that hydrogen atom reacts with the chlorine molecule to give you the converse intermediate product to this, which immediately breaks down into chlorinc atoms plus the hydrochloric acid, $H + Cl_2 = HCl_2$ and $HCl_2 = HCl + Cl$. You may say this is all uncertain theory, and is no more certain than anything we have had before, but that is not at all the case. Through the use of Prof. Nernst's heat theorem, he has been able to calculate the actual heat reaction of these two intermediate reactions. They both come out

positive with very high values, something of the order of 20,000 small calories in both cases. That means that these two reactions will both take place spontaneously so far as the chemical free energy goes and so far as what we know about the action of atoms in reactions, we would expect them both to be instantaneous reactions from the standpoint of ordinary chemical kinetics. You can see that all you need to explain the photo-chemical reaction is at hand, and all you need is some form of radiant energy that will produce an initial splitting of either one of these molecules, it doesn't matter which, and then the reaction will proceed spontaneously and will very far exceed, of course, the requirements of the Einstein photo-chemical theory. It comes out about the same as we would expect in the ionization of gases by any ionizing agent like radium rays, and it may interest you to know that this same reaction, which is very phenomenal from the standpoint of light, is also exceptional for these other sources of energy, and out of 15 or 20 reactions which have been examined under radium rays, it appears that in all cases the amount of chemical action taking place is of about the same order very closely as the total ionization produced in the gas, that is, the actual number of pairs of positive and negative charges. That is extremely important, because the two things might have differed by several million-fold in either direction as far as we could anticipate. So here you have this important photo-chemical reaction, which shows the same abnormality as Dr. Taylor and Prof. Bodenstein showed for the *alpha* rays, and which I had occasion to calculate earlier for the *beta* rays from other sources. What is actually found then is that the number of molecules of hydrogen chloride formed from one original atom, split from either one of these atoms, is of the order of several thousand; that is, you only need one initial atom split off to set up a reaction which goes spontaneously to combine about four to six thousand. The question occurs to you at once, "Why stop there? Why not go on indefinitely after splitting off one atom?" The answer is very simple, that although the concentration at any time of hydrogen atoms and chlorine atoms is extremely small, you will have some chance of their getting together directly, which would stop the reaction. The reaction would therefore come to an end; soon after you cut off the source of light, so that is why it is necessary

to keep up the source of radiant energy. The other means of removing the free atoms besides the straight cross reaction is by some other substance which can react with them to remove them from the field of action, and oxygen is one of those substances. That is where the inhibitors come in. It is well known that a small amount of oxygen inhibits this reaction or will prevent it from starting at all; so we can explain the whole thing without the assumption of any unusual effects not generally recognized by chemists. I do not mean to convey the impression that I do not think unusual effects may not apply in the case of contact catalysis. I hope we will proceed soon to get a solution of that also. We used to think of atoms as little round bullets almost devoid of properties; it was almost impossible to get anywhere in theorizing on the subject of kinetics or catalysts. Now we have learned that the structure of the atom is extremely complex, that the atom is a whole solar system of electrons revolving around the atom, and we have such a multitude of problems presented to us that it is very easy to make all kinds of theories, but it may be very difficult to decide or prove which is correct. It has been estimated that it may take about a hundred years to work out entirely and get experimental proof of what the structure of the atom is. I hope it will not take that long, because I am afraid that few of us would live to see it through; I would like to see the subject of catalysis cleared up very much earlier than that.

CHARLES L. PARSONS[2]: I know too little about catalysis to attempt to take part in any real discussion of the subject, and I have not imagination enough to present any new theories, but there are one or two points that I should like to bring out. First, I thought that Dr. Taylor was coming to a point which, if I heard correctly, he did not come to, namely, that silica may, like carbon, exist in two forms, and that we have not yet found the other form of silica. There are two forms of carbon, one active and the other inactive; one of them acts as a catalyzer; the other does not. It may be that silica, which resembles carbon as closely as any other element, may also have an active form which has not yet been found. The inactive carbon, I believe, does not have very great adsorptive power, while the active does. The silica gel does have reasonably high adsorptive power, but not com-

[2] Secretary of the American Chemical Society, Washington, D. C.

parable to carbon; if we really get an active form it may have a higher adsorptive power and may possibly act as a catalyzer. I think Dr. Taylor said that while catalysis did not always follow adsorption, adsorption in his opinion preceded catalysis. I am not denying that it may be true, but I find it hard to conceive it to be true, especially in the case of the catalytic action of platinum on the reaction between ammonia and oxygen. The higher the temperature, the more readily that reaction takes place, at least within limits; we know it to be so up to 1050°; also the capacity increases with the temperature, so that at 1025° a mixture of ammonia and air can be passed over a platinum gauze at the rate of about 40 cubic feet (1.15 cub. m.) a minute and the reaction takes place just as fast as you can pass it, up to that limit; whether it has a higher limit or not we do not yet know. Now I find it very hard to believe that there is any question of adsorption at that rate of flow of gas over a platinum wire.

ANDREW EGAN: The views I have to give are not my own, but are taken from a recent article by a Frenchman, John Perrin, in a recent publication. To start with, he proposes a new hypothesis for explaining all chemical reactions, which are supposed to be brought about through the influence of photo-chemical radiations. In order to get the molecule into proper condition a certain frequency is required, whether it is ultra-violet or red; it requires action on the particular structure of molecule. It must be hit by the right frequency of radiation, and then changes into another substance, and in doing so it gives out another kind of radiation. He uses this theory in reference to catalysis; the catalytic reagent forms a new substance with the original material unchanged, and the most of such reactions follow probably this way; $A + B$ gives first AB; that changes over to $A' B$, which finally splits down into the final compound, $A' + B$. By taking each step and calculating the frequency and the chemical changes, we can get a ratio of the activity frequencies to the reaction velocity. The ratio of the frequency of the radiation which starts the substance to reacting to μ', the frequency of the radiation produced when the product is formed, is equal to $d \log. K$ divided by $d \log K'$, K and K' being the velocity constants in these two reactions, in this reaction and the reverse. This same relation also applies for the reaction at two different temperatures.

If we take the temperature T and the temperature T', then the frequency required to produce the reaction at the temperature T is to the frequency required to produce it at T' as the logarithm of the velocity constant at T is to the logarithm of the velocity constant at T'. This shows us that if a catalyst tends to decrease the thermal acceleration it decreases the active frequency also. Take the case of the action of chromic oxide on formic acid, which is catalyzed by sulphuric acid. A change in temperature from 20° to 30° produces a ratio of these constants equal to 1.16. That means that the wave length has been increased proportionately. Dahr has supposed this is a general rule, supported by many cases, that where the acceleration is increased an increase in temperature decreases the rate of acceleration. Suppose the catalytic action cuts the acceleration in two; the wave length is changed from μ to 2μ, which at present is very much greater intensity at the lower temperature.

H. S. Taylor: There is just one point in this connection, whether adsorption can take place, and I think it is a rather interesting experiment. Take a strutt tube filled with nitrogen and pass an electrodeless discharge through; you have got an active form of nitrogen, say the ozonized form of nitrogen; that gradually changes back into ordinary nitrogen, and while that is occurring the bulb luminesces; it is a beautiful glow in the bulb while this change back from active to ordinary nitrogen is taking place, and in an experiment in the Royal Institution in London by Bone that disappearance occupied about a minute and a half. Now he did the experiment this way: into the bulb he put a tiny capillary tube in which he placed a length of copper oxide wire; he passed the electrodeless discharge through, and immediately the glow started. He tipped his bulb up and dropped the copper oxide wire just through the bulb and immediately the glow finished. I mean to say that the rapidity with which all those molecules rushed to that copper oxide wire was practically instantaneous; a reaction which ordinarily took a minute and a half was instantaneous immediately it comes in contact with some substance there. Then there was one other point raised by Dr. Lynn which throws some light on a case which Dr. Bancroft brought up earlier. It is just this: Dr. Lynn points out that these catalysts are all accelerating reactions which of themselves are tending to take place. In other

words, they are promoting the equilibrium which would normally be established. Take this particular case, the case of ethyl alcohol to give ethylene and water; Dr. Bancroft pointed out that in actual practice they added an excess of steam, and he said that was rather surprising because you would expect that that would spoil the formation of ethylene. It would not spoil it if the normal equilibrium constant of the reaction,

$$K = \frac{(C_2H_4)(H_2O)}{(C_2H_5OH)}$$

was infinitely large; if that was exceedingly large, the addition of a thousand times the excess of water vapor would practically not affect the equilibrium constant at all, and when the manufacturers added the steam, they also put up the temperature; in other words, they also went over to the conditions under which alcohol will decompose to ethylene and water; and this is my view of what happens, that when the alcohol molecules strike the particle of kaolin, it may come off in two different ways, as alcohol or as water vapor and ethylene; the possibility of its coming off as alcohol is greater the lower the temperature; the probability of its coming off in a different way, namely the water vapor staying momentarily behind the part of the molecule which is flying off, namely, ethylene, increases with increasing temperature. It depends on the temperature, the probability of the molecule coming back from the surface as C_2H_5OH or the C_2H_4 setting out on its independent existence and leaving the water vapor to evaporate from the surface.

Colin G. Fink[3]: I should like to add to Dr. Taylor's comment on the velocity of adsorption that I made measurements[4] on the velocity of adsorption of SO_3 in the contact process and by all methods available, but I could not get any definite values. The velocity was, as far as a chemist can regard it, an infinite velocity. As soon as the SO_3 was in the presence of the platinum, it was adsorbed; the SO_3 could not be introduced into the reaction chamber without being adsorbed instantly. We must revise our ideas of adsorption as compared with the popular idea of absorption; the absorption of water by a sponge is at a comparatively slow

[3] Head of Research Laboratory, Chile Exploration Co., New York City.
[4] Zeit. physikal. Chemie (1907), 60, 1-54.

rate, it corresponds to a slow rise in a capillary tube. There are two entirely different phenomena, a very rapid one and a comparatively slow one. I think the distinction is brought out very nicely in Mr. Chaney's paper; there is a very rapid adsorption in the first instance and then a very slow, what MacBain of Bristol, England, calls "sorption" thereafter.

CARL HERING[5]: May I call attention to a point which does not seem to have been mentioned. When a catalyzer adsorbs a gas it means (and we must admit it whether we want to or not) that that gas has then been changed physically into what we ordinarily call a solid or at least a liquid. We know that when that change takes place energy is set free. We also have heard from Dr. Taylor that most of the catalyzers are of a spongy nature, that is, the mass is small. The heat set free by the condensation of a gas to a solid involves two latent heats; the amount of it might be small and yet raise the thin and relatively small surface of the catalyzer to quite a high temperature, and perhaps that temperature, although it may be only momentary, might affect the chemical reactions. Some years ago I showed before this society how steel can be melted under water simply by generating a very large amount of heat very suddenly on a relatively small surface of the immersed steel.

It may be maintained that the gases after being condensed on a catalyzer again pass off as gases, and that therefore they would in this second operation consume this heat again. But as a great many processes in nature are of an oscillating character, might it not be possible that this process is also of an oscillating character, and that the heat which must be set free in the catalyzer when the gas condenses, may be that necessary to start the chemical reaction, as a spark does in a mixture of hydrogen and oxygen.

N. K. CHANEY[6] (*Communicated*): Prof. Taylor has called attention to the change in the rates of adsorption of any given gas when the surface of the charcoal has been "cleaned" by washing it repeatedly with the gas which is to be adsorbed. The phenomena which Dr. Harned noted in the case of chlorpicric absorption has been shown also by Dr. Harkins to hold for benzene

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[6] Asst. Director Research and Development Laboratories, National Carbon Co., Inc., Cleveland, Ohio.

absorption. That is the r a t e of benzene absorption increased with each successive adsorption and removal of the same.

Yet the significant thing is that the a m o u n t of gas absorbed when saturation is reached is not materially altered. It does not seem to me that Prof. Taylor's explanation of the mechanism of this increased rate being in any way related to the number of spaces or amount of free space on the charcoal is tenable. For when you come to think of it there is no reason to believe that the charcoal "washed" with a given kind of gas is any "cleaner" than it was before. The difference is merely in the kind of molecules absorbed on the surface. This brings out the significant fact that adsorption is most rapid when the residual molecular film on the carbon surface is exactly the same as the gas to be absorbed, and that the presence of any other kind of molecules even in minute amounts enormously lessens the i n i t i a l rate of absorption. This suggests among other things a simple analogy to the lessening of the diffusion rate of a gas in the presence of another gas. Into a perfect vacuum the diffusion of a single gas is practically instantaneous. The situation is very different if it must pass through a layer of another gas already present. This is sharply illustrated by the difference in time which it takes to saturate charcoal in a closed vessel with a gas whose partial pressure is held constant. The degree to which air as inert gas is evacuated from the vessel determines the rate at which equilibrium ensues between the adsorbed gas and the charcoal. And this rate may vary from a week at atmospheric pressure to a few minutes at moderately low pressures. I am not sure that the neutral molecules do not in effect set up a diffusion layer, whose interference is largely responsible for the retardation in absorption of the gas under consideration. We know both from Dr. Semon's observations and my own that the adsorbed layer is not absolutely fixed but tends toward equilibrium with a definite partial pressure of the same gas. Whether there is any further significance than this in the fact that absorption is most rapid when the residual layer is identical in kind with the gas being absorbed, has yet to be shown.

H. S. Taylor (*Communicated*): Since this paper was published certain experimental results obtained in the author's laboratory seem to indicate that not only the velocity of adsorption but

also the velocity of de-sorption is influenced by the presence of foreign gases. Both factors, in the author's view, must influence the velocity of catalytic action.

In reply to Dr. Hering it may be observed that certain equilibrium reactions when catalyzed give equilibrium constants identical with those obtained in the simple uncatalyzed gas reaction. If the increased reaction velocity were due to increased temperature at the point of contact, would not divergent values be found for the equilibrium constant in the two cases?

Presented as part of a Symposium on "Catalysis" at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 26, 1919, H. S. Taylor in the Chair.

THE THERMAL PROBLEM IN ORGANIC CONTACT CATALYSIS.[1]

By Wilbert J. Huff.[2]

As a part of its work in the utilization and conservation of our mineral resources, the United States Bureau of Mines is interested in some catalytic and quasi-catalytic contact reactions and their attendant thermal problems. Among such reactions are the cracking of petroleum, and the chlorination and hydrogenation of hydrocarbons.

Heat effects accompany all chemical reactions, so the transference of heat is ever a problem, and one especially important in most large scale productions. Theoretically, three classes of reactions can be distinguished, reactions whose heat is small, reactions markedly endothermic and reactions markedly exothermic. Since, however, most contact catalyses take place rapidly only at elevated temperatures, in this discussion only two cases will be distinguished—reactions to which heat must be supplied and reactions from which heat must be absorbed. Throughout, however, the reactions discussed are used merely as illustrations of methods of procedure, and the figures given merely examples, not specific data, while the whole is intended to present a few general problems, and not a complete survey of the entire field of thermal relations involved in contact catalysis.

REACTIONS TO WHICH HEAT MUST BE SUPPLIED.

The Rittman Process.

In including the vapor phase process for the cracking of petroleum in a discussion of contact catalytic reactions, the writer is aware of the doubts and complications which surround the exact chemistry of this process. Nevertheless, it can not be doubted

[1]Published by permission of the Director, U. S. Bureau of Mines. Manuscript received September 20, 1919.

[2] Research Chemist, U. S. Bureau of Mines, Pittsburgh, Pa.

that catalytic or quasi-catalytic surface effects take a part in this process and that the importance of this part can hardly be overestimated.

Moreover, while some endothermic and some exothermic reactions undoubtedly take part simultaneously,[3-4] experience shows that heat must be supplied to the total.

Fig. 1 shows a laboratory furnace for the Rittman process. The oil is fed from reservoir *a* through pipe *b* upon chain *h* suspended within the furnace tube *e*. It is vaporized and cracked by heat supplied by electric resistance *f*, then passes out and is condensed through condenser *q*. The temperature of the contact wall *e* is measured by the thermocouple peened into the wall in the manner and at the point shown. If desired, another thermocouple may be inserted through *p* to measure the vapor temperatures.

We will arbitrarily avoid a discussion of the heat of reaction and turn our attention to the physical side of the thermal problem and the resulting influence upon the chemical reactions. Heat must be supplied to bring the liquid oil to the temperature of vaporization, to cause the change of state, and to raise the temperature of the vapor so much that the active surface is maintained at the optimum temperature for gasoline production without excessive gas formation. This entails a considerable absorption of heat in the first part of the system. Moreover, this absorption is constant only with a constant flow of oil of constant chemical composition at a constant pressure. Variations in any of these will obviously vary the amount of heat absorbed here, although, of course, some of the variations will probably be found upon analysis to be so small that they may be disregarded. Unfortunately the analysis of the information available will show much data lacking on the specific heats and heats of vaporization in question—a lack that is often encountered in problems dealing with thermal relations in organic chemistry. Heat must be supplied to compensate for other losses—radiation and convection losses from the metal parts at the exposed ends of the furnace tube and losses through the insulation *g*. When the heat is supplied through a resistance coil of equal pitch (as is done here). the losses at the exposed ends are important, for they alone inevitably cause temperature differences at different points on the

[3-4] For these and following numbers see references appended to this paper.

contact wall—thus the temperature at the thermocouple junction shown is undoubtedly higher than at points opposite the lower and upper ends of the resistance wire. The differences in temperature between these various points will depend among other things upon temperature differences between the furnace and the room (a greater difference giving a steeper temperature gradient), upon the rate of flow of the hydrocarbons within the system and their chemical composition (for these assist materially in the distribution of heat along the furnace wall), upon the pressure within the system (for the heat capacity of the vapors per unit volume changes with the pressure), and upon the depth and thermal properties of the carbon which separates upon the contact surface.

A thoughtful consideration of these factors will show that although a careful study of one invariant set of conditions might enable one to design such a furnace so that it is capable of maintaining the same temperature throughout when subjected to this invariant set of conditions, the design would not be of great value, for the uniformity would be destroyed as soon as one of the conditions is altered.

Were the desired light oils comprising gasoline stable end products of the reaction under operating conditions, a difference in temperature on the contact surfaces would not be a very serious matter. Unfortunately, this is not the case. The vapors of the light oil produced by the cracking of the heavy distillates themselves undergo thermal decomposition to still other products, some of which are gases at ordinary temperatures and pressures. The process, therefore, depends upon an adjustment of conditions to attain an approximation to the ideal condition for as rapid as possible a production of light oils without an excessive production of gas. This, of course, renders necessary a contact temperature as nearly uniform as possible. The writer is told by Dr. E. W. Dean, who has kindly supplied the figure shown and the information concerning the process, that the effective temperature range for the commercial production of gasoline is only about 30 degrees Centigrade (from about 550 to about 580° C. for Pennsylvania oil) and that the gasoline production follows the temperature recorded on the peened in thermocouple *i* and not that of the couple exposed to the vapors, thus showing that surface action does play a leading part.

The advantages of the furnace of Fig. 1 lie in its simplicity and reproductibility, both important merits. Its thermal disadvantages can, of course, be eliminated to some extent by other designs which, however, introduce some complications.

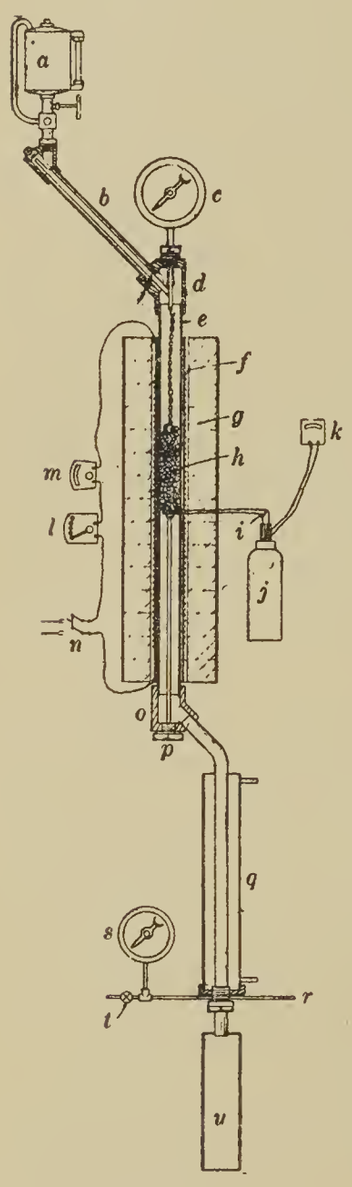

FIG. 1.

The principles upon which such designs may be based will no doubt suggest themselves to the reader of the illustrations of other processes following.

Catalytic Dehydration of Alcohol Upon Aluminum Oxide, Clay, etc.

Of recent interest in connection with the "mustard gas" synthesis is a well-known reaction to which heat must be supplied, the catalytic dehydration of alcohol to ethylene upon aluminum oxide, clay, etc. Here fortunately the end product is comparatively stable, so the thermal problems are not so annoying. The catalytic surface is a porous contact mass such as is prepared by coating pumice with aluminum oxide, or by forming clay into pellets. This may be shoveled in long reaction tubes many inches in diameter. The rough diagram (Fig. 2) may be taken to represent the cross section of the entering portion of some such a tube. If the necessary heat is supplied through the external diameter of the tube as indicated, the porosity of the catalyst parts, their poor thermal contact with one another, and the resulting numerous "dead gas films" which thus interpose render it inevitable that temperature differences intervene between the periphery *aa'* and the center *b* of the contact mass near the inlet end. Inequalities of temperature on the exterior surface of the tube, inequalities in thermal conductivity between various parts of the contact mass, together with radial and eddy currents prevent the isotherms of practice from following any such simple lines as *aca'* but there is

undoubtedly an irregular volume of lower average temperature which we may for simplicity in this discussion designate as *aa'c* in contrast with volume *dd'a'ca*. This is the more serious the greater the diameter of the tube, other things being equal. Since alcohol vapor occupies some thousand times greater volume at the more favorable temperatures of reaction than it does in a liquid form at ordinary temperatures, small cross section areas are not desirable in large scale production. The magnitude of the average temperature differences can of course be diminished by preheating the alcohol vapor, but because at high temperatures most metallic surfaces catalyze the decomposition of alcohol in an undesirable manner, because gases change temperature largely by convection processes, and because gases at high temperature possess low heat

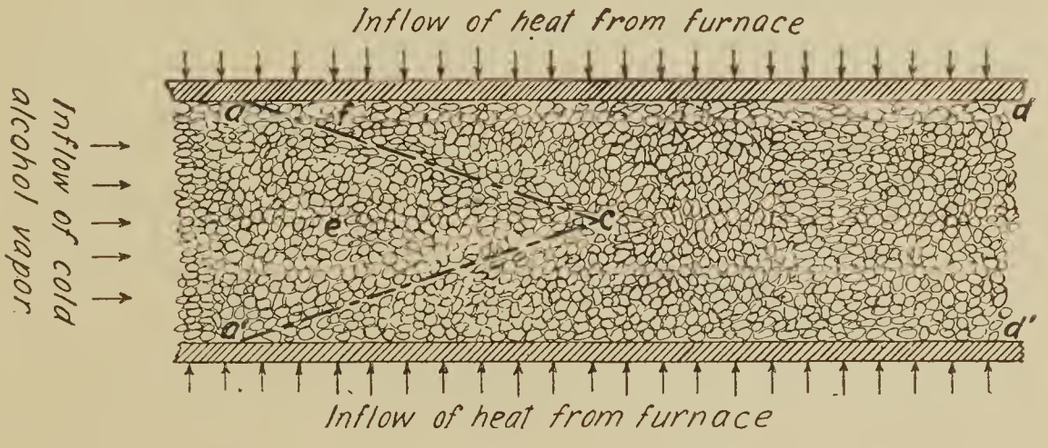

FIG. 2.

capacities per unit volume, it is preferable to preheat only to a certain point, the remaining temperature change being brought about by absorption of heat from the catalyst itself. However, if the tube is sufficiently long it is evident that all of the vapor must ultimately make proper contact at a temperature sufficiently elevated to insure completion of the reaction. If a long tube is adopted, however, it is evident that an ethylene molecule from a completed reaction in the neighborhood of *f* must be subjected to many more contacts and a longer period of thermal treatment because of the presence of the undecomposed alcohol molecule *e* in the colder zone. Yields of 90 percent and over in such tubes are only possible because the further decomposition of the ethylene possesses only a slow reaction velocity under the conditions used.

To maintain a large cross section area, a uniform temperature

and a contact not too prolonged, an electrically heated cake or grid such as that shown in Fig. 3 possesses many advantages. This figure was taken from an unpublished report by G. A. Hulett and co-workers.

The chief difficulty in the ethylene process is the deterioration of the activity of the catalyst, which gradually becomes coated with tarry or carbonaceous matter. Molecular condensations may also take place in the catalyst.[5]

The writer understands that the ethylene furnace at Edgewood consists essentially of two concentric tubes, with the catalyst charged in the annular space between. Such an arrangement reduces the temperature differences between the exterior and interior of the contact mass.[20] Thus, a combination of an eight

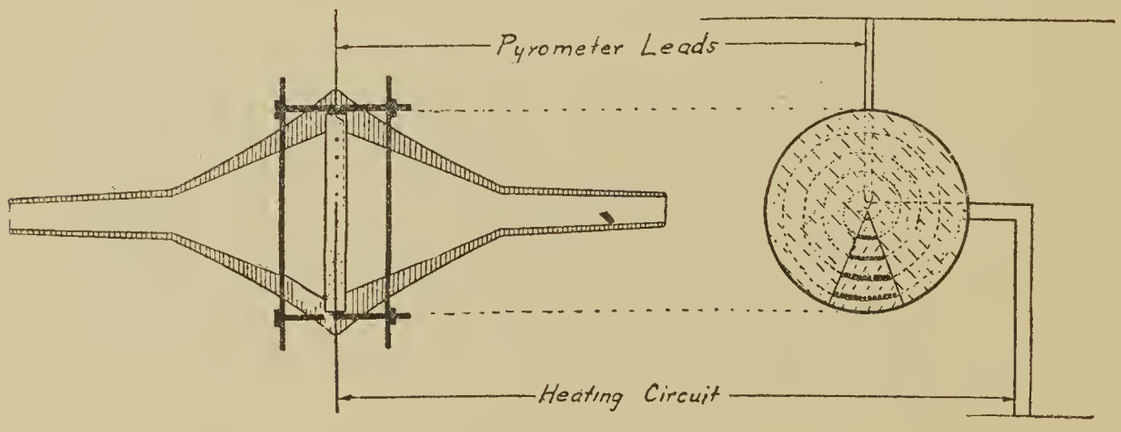

FIG. 3.

and a ten inch tube will give the same available cross section as does a completely filled six inch tube, yet in the concentric tubes no point on the catalyst is farther than an inch from the outside walls, while in the six inch tube points along the axis are three inches from the outside walls.

At the Badische ethylene plant the catalyst tubes were copper of small cross section, surrounded by molten potassium nitrate. The catalyst was aluminum oxide.[19]

In the foregoing discussions mention of the functions of heats of reaction was arbitrarily avoided. The purpose was to illustrate how heat capacities, latent heats, thermal conductivities and reaction velocities may be involved in problems of contact organic catalysis, and especially to illustrate how the maintenance of a contact surface at the same temperature throughout may present problems even when heats of reaction are not considered, and

how in consequence the stability of the desired product is of importance. (In practice, it is never safe to disregard heats of reaction. Thermal losses due to radiation or convection vary widely with different types and sizes of apparatus, and in consequence heats of reaction negligible under some conditions may be very important under others.)

When to these conditions is added an endothermic reaction, the differences resulting are mainly only of degree. Since such reactions proceed with the absorption of energy, the maximum temperature will not exceed that supplied externally. Moreover, momentary local superheating entails locally increased reaction velocity and consequently locally increased absorption of heat, so the reaction itself serves, figuratively speaking, as a thermal balance wheel. Provided the desired product be stable enough to permit the use of a long reaction tube, an endothermic contact catalysis presents no impossible problems in construction and operation.

The problems offered by exothermic reactions, however, are not so simply dismissed. These are considered in the following pages.

REACTIONS FROM WHICH HEAT MUST BE ABSORBED.

Partial Combustion Processes—Preparation of Phthalic Acid from Naphthalene.

Partial combustions to intermediate products of value afford some of our most interesting examples of contact catalyses from which heat must be absorbed. A process of this kind is a commercial synthesis of formaldehyde. More novel is the recent synthesis of phthalic acid and other valuable products by the interaction of air and naphthalene vapor over oxides of vanadium and molybdenum.[6] It does not detract in the least from the merit of the inventors of this process to point out that the possibilities of this field were indicated as early as in 1892 by J. Walter,[7] who after describing experiments on the partial combustion of toluol, phenol and some aromatic amines over catalyzers, one of which was vanadium oxide asbestos, concluded that such processes are of no practical use with an expensive raw material where a small loss due to complete combustion outweighs the oxidation cost, but that the case is quite different when there is a great difference

between the market value of the raw material and the intermediate oxidation product. Among such possible raw materials he cited toluol, naphthalene, natural methane, and low boiling petroleum distillates. There is, however, no record that Walter ever attempted to adopt his own suggestion with regard to naphthalene, so to Gibbs and Conover goes the honor of discovering what the writer considers one of the most interesting processes in organic contact catalysis.

Surely no one could have asked for a more encouraging differential than that existing for this process at the time the specifications were filed—phthalic anhydride at not less than $3.50 a pound from air and naphthalene at nine or ten cents a pound. Moreover, the process is continuous and when once begun proceeds with the liberation of energy. This promises a low labor charge and low heat expense, so only a small percentage yield would enable the process to compete profitably with pre-war imported phthalic acid at some twenty-six cents a pound from naphthalene at some two and one-half cents a pound. It is not surprising then that the patentors soon sought co-operation in the developments designed to lead to large scale manufacture.

One of the largest, if not the largest problem in such development must have been the proper absorption of the heat of reaction, as a glance at available thermal data and a few simple calculations will readily indicate.

One mole of solid naphthalene burned completely to carbon dioxide and liquid water gives out some 1230 kilogram calories. Solid phthalic anhydride similarly gives about 780 kilogram calories. It follows then that the heat of formation of solid phthalic anhydride with the production of liquid water and gaseous CO_2 from naphthalene and oxygen is about 450 kilogram calories. The writer does not have the specific heat of naphthalene vapor, or of phthalic anhydride vapor, nor the latent heats of fusion and vaporization of phthalic anhydride and cannot, therefore, calculate the heat of reaction at the temperature stated to be the most desirable (500° C.). For the purpose of illustration, 450 kilogram calories will be taken as a rough approximation. The reaction theoretically requires 4.5 volumes of oxygen or about 22.5 volumes of air to one volume of naphthalene vapor. In one of the patents an excess of oxygen, preferably four times the theoretical is

recommended. In that case, the reaction gases consist of about ninety volumes of air to one of naphthalene. Taking the molecular heats of nitrogen and oxygen as 7 and the specific heat of naphthalene vapor equal to that of benzol at 350° C., it follows that the complete conversion of naphthalene to phthalic acid in a perfectly insulated space would raise the temperature of the mixture about

$$\frac{450{,}000}{90\ (7) + 128\ (0.5)} = 648°\ \text{C.}$$

Should naphthalene vapor possess a specific heat twice that assumed, the temperature would still be raised 594° C. In the catalytic chamber heat will be expended in raising the temperature not only of the gases but also of the catalyst, its mounting and chamber, while some will be lost through radiation or convection. Under constant conditions, however, the temperature attained at equilibrium will depend only upon the amount of heat produced by the chemical reaction, the amount of heat absorbed by the incoming gases, and that lost through radiation or convection. The radiation or convection losses are characteristic of the apparatus and are functions of temperature differentials, so it is difficult to assign one general probable value to these losses. Five percent has been suggested as a basis for a similar discussion in the ammonia oxidation,[8] and since that is probably as good as any other, it will be used for illustration here. Applying it to the case under consideration we see that the complete reaction within the catalyst chamber would raise the temperature of the contact mass either 616 or 564° C., depending upon which of the two assumed values for the specific heat is taken as correct. Given then an apparatus whose thermal losses average about five percent over the ranges of temperature experienced, whose catalytic surface is sufficiently extended to insure complete reaction at even the lowest reaction temperature, disregard the by-reactions, save the complete combustion of phthalic anhydride, and supply from the outside independently and continuously enough heat to maintain the system, gases and all at 250° C. (the starting point of the reaction), then turn in the above reaction mixture. Undoubtedly the temperature will rise to red heat, the combustion of phthalic acid to carbon dioxide and water and the attendant liberation of some 1230 instead of 450 kilogram calories will go on apace as

the upper temperature limits of the reaction (650° C.) are approached and passed, until the oxidation proceeds not only upon the catalytic surface but throughout the entire gaseous mass, while the oxide catalyst will probably sinter or undergo molecular transformation or both, so that its catalytic properties are seriously impaired or completely lost; in short, the whole process will produce carbon dioxide, water, heat, and trouble.

This prediction is given only to illustrate the general case. Perhaps analogous conditions were encountered in the actual development of the process, perhaps not. Consideration of the poor thermal conductivities of oxides, of pumice, of asbestos, clay, or

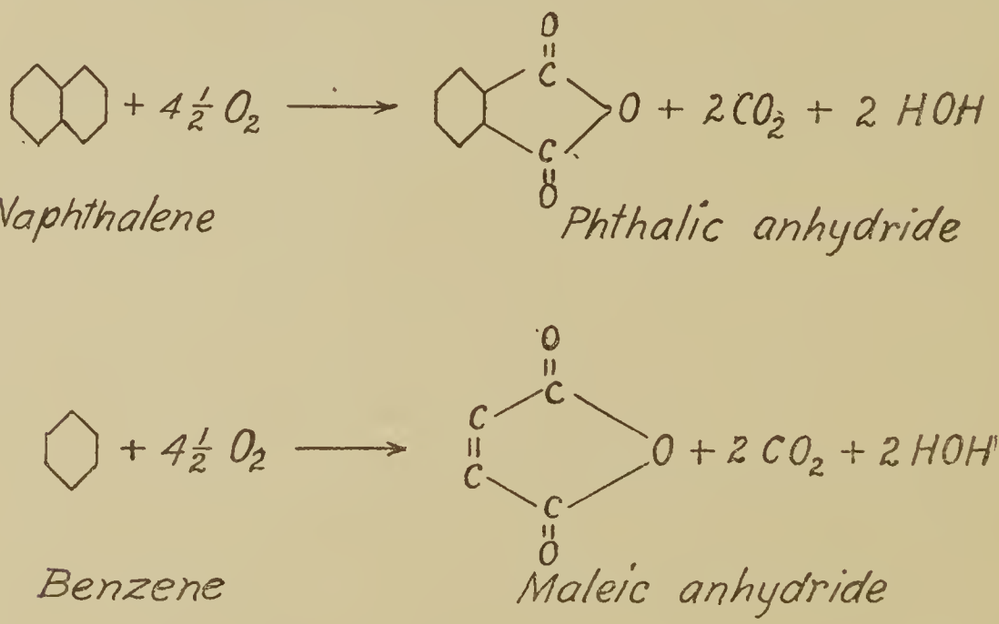

FIG. 4.

other mounting material, of the unequal and irregular thermal contacts between portions of the catalyst mass, of the numerous "dead gas films," of many other factors leading to temperature differences throughout the contact mass, of local overheatings which increase local reaction velocities, which in turn again increase overheatings, of the difficulties which beset the successful initiation of the reaction without overstepping the desired temperature—such considerations all lead the writer to the conclusion already stated, namely, that one of the largest problems in the development of such a process must be the proper absorption of the heat of reaction.

Much that has been said about the foregoing process could be said about similar partial combustion processes for other oxygen compounds.

Maleic Acid Process.

Thus a glance at the symbols for benzene and naphthalene, together with those of maleic and phthalic anhydrides (see Fig. 4) indicates at once the analogies which might exist between the two processes, so it is not surprising that the same catalyst, vanadium oxide, should be found in both.[9]

Analogous thermal problems are also to be expected.

The maleic acid process is especially interesting because of the possibilities which it offers. This acid, containing as it does the reactive ethylene linking, may be oxidized to tartaric, reduced to succinic, or hydrated to malic acids. The spread of prohibition is diminishing the tartaric acid supply so a new source or a new substitute will be quite welcome. Processes for the synthesis of dyes and resins using succinic acid are known, but little used on account of the high cost of the material from past sources. Reactions for malic acid also offer possibilities for development.

Other Partial Combustion Possibilities.

To indicate a few of the additional possibilities of partial combustion, some preliminary experiments by the writer may be not without interest.

The passage of a mixture of air and an excess of kerosene vapor of unknown origin over a short catalyst of vanadium oxide at temperatures which varied from about 350 to about 500° C. (temperatures recorded on a thermocouple imbedded in the center of the catalyst) gave a condensate in two layers, one of which was orange brown in color and contained largely kerosene, while the other was water strongly acid in reaction. The whole smelled strongly of aldehydes, one of which was probably acrolein, for the water extract gave many of the color tests for this compound. Upon evaporation the water layer deposited a sticky, resinous mass. Distillation of this gave needles of an acid compound, soluble in water and in benzol, but insoluble in petroleum ether. The melting point varied over a wide range. Attempts to crystallize this to obtain a satisfactory melting point were without success. Finally strongly nitric acid was tried. From this medium were obtained pale yellow crystals melting at 184 to 185° C. (uncorrected). Recrystallization from hydrochloric acid gave a melting point of 190° C. Further recrystallizations were not made. The

crystals from the nitric acid solution gave a neutralization equivalent of 86 and sublimed without discernible decomposition to long needles resembling those of succinic anhydride. Condensation with paratoluidine following exactly the directions of Mulliken for succinic acid[10] gave a toluide whose melting point was 198° C. Further recrystallizations from alcohol did not change this melting point. While this datum is not sufficient for absolute identification, the small quantity of material did not permit the determination of

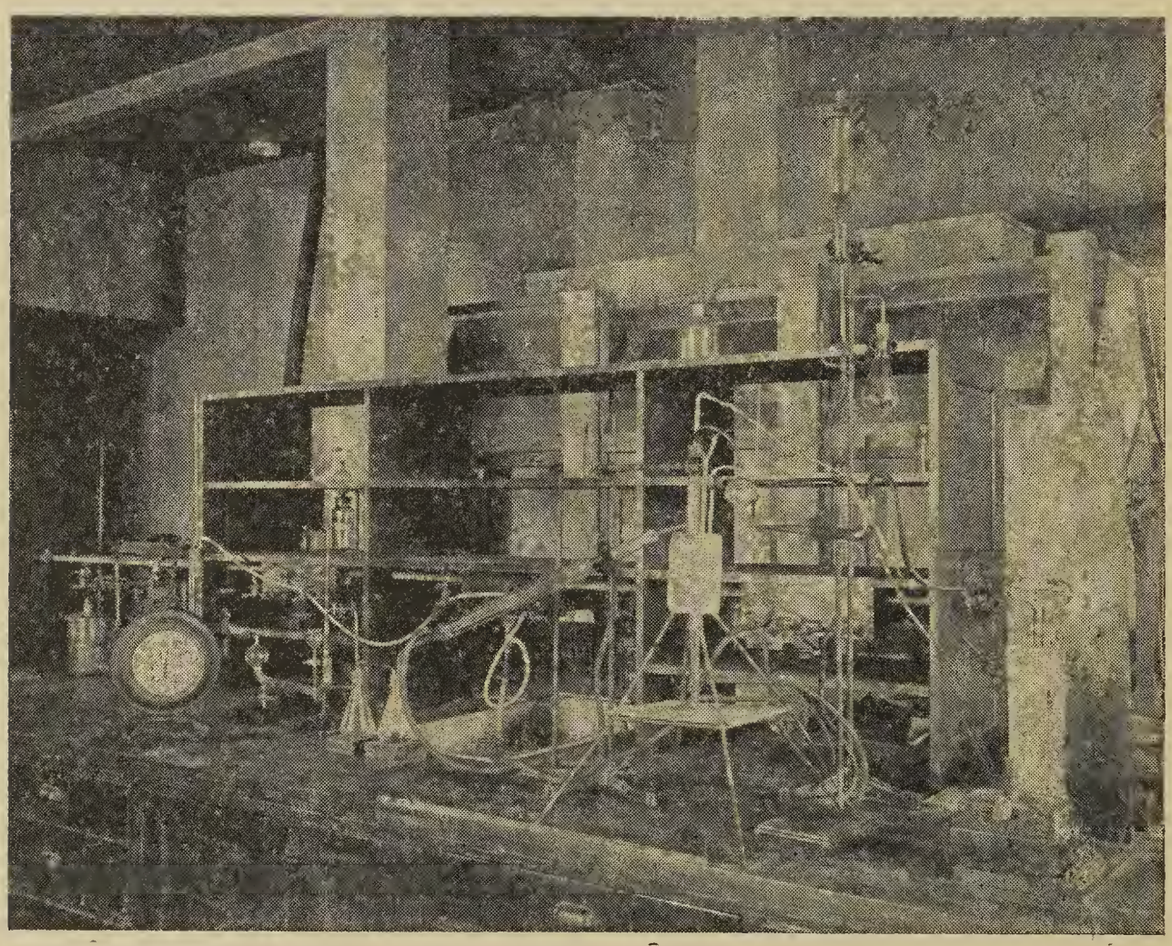

FIG. 5.

[For legend see end of paper.]

more. Probably the acid is tetramethyl succinic, whose neutralization equivalent is 87, whose melting point is 195, but the melting point of whose para-toluide the writer has not yet ascertained.

Preliminary experiments were made to throw light upon the mechanism of the reaction. The evidence so far accumulated, while not complete, indicates that at temperatures above 350° C. long chain hydrocarbons of the paraffin series burn over vanadium oxide to oxides of carbon and water, producing at intermediate

stages aldehydes and acids whose existence must be ephemeral for only traces are found in the condensate. This is of course in keeping with the findings of Bone and his co-workers[11] on the mode of the combustion of methane and ethane, and of Sabatier and Mailhe[12], using oxygen and other catalyzers.

Attempts to prepare the above described acid from an authentic sample of Pennsylvania kerosene were without success.

With naphthenes, however, the possibilities appear to be greater. Experiments with cyclohexane, previously freed from benzene by nitration, gave appreciable quantities of maleic acid, as well as water, oxides of carbon, and some aldehydes.

This evidence, coupled with that already cited on the oxidation of benzene and naphthalene suggests that the rupture of a hydrocarbon ring by partial combustion over vanadium oxide under controlled temperature conditions is succeeded by the production of two carboxy-acid groups which may, in some cases, form ring anhydrides whose survival is much aided by the well-known stability of certain types of ring compounds.

Mention of these preliminary experiments is made here merely to suggest some of the possibilities for development which await the practical solution of the thermal problems involved in contact catalyses developing large heats of reaction. Of course naphthenes possess greater heats of combustion than do aromatics of like carbon content. Moreover, their thermal decomposition gives some straight chain compounds.[13] Their partial combustion should therefore offer greater thermal problems.

Even the laboratory investigation of these oxidation catalyses offer thermal problems not to be disregarded, for the usual types of heating apparatus, such as gas combustion furnaces or asbestos jacketed and electrically heated tubes are practically worthless. An excellent expedient consists in surrounding the catalyst tube with a medium having a high heat capacity such as a molten sodium-potassium nitrate mixture or a large block of metal, so that the development of large amounts of heat will cause only small and therefore controllable temperature changes in the catalyst. Since the thermal interchange from the center to the periphery of a contact mass consisting largely of pumice or of asbestos is comparatively poor, the experimental catalyst tube should not have too great a diameter, say not over a maximum of one inch.

It is also desirable to increase the heat capacities of the incoming gases if possible. This may conveniently be done either by using an excess of one factor or by admixing the factors with an inert substance. For this reason, air is preferable to oxygen.

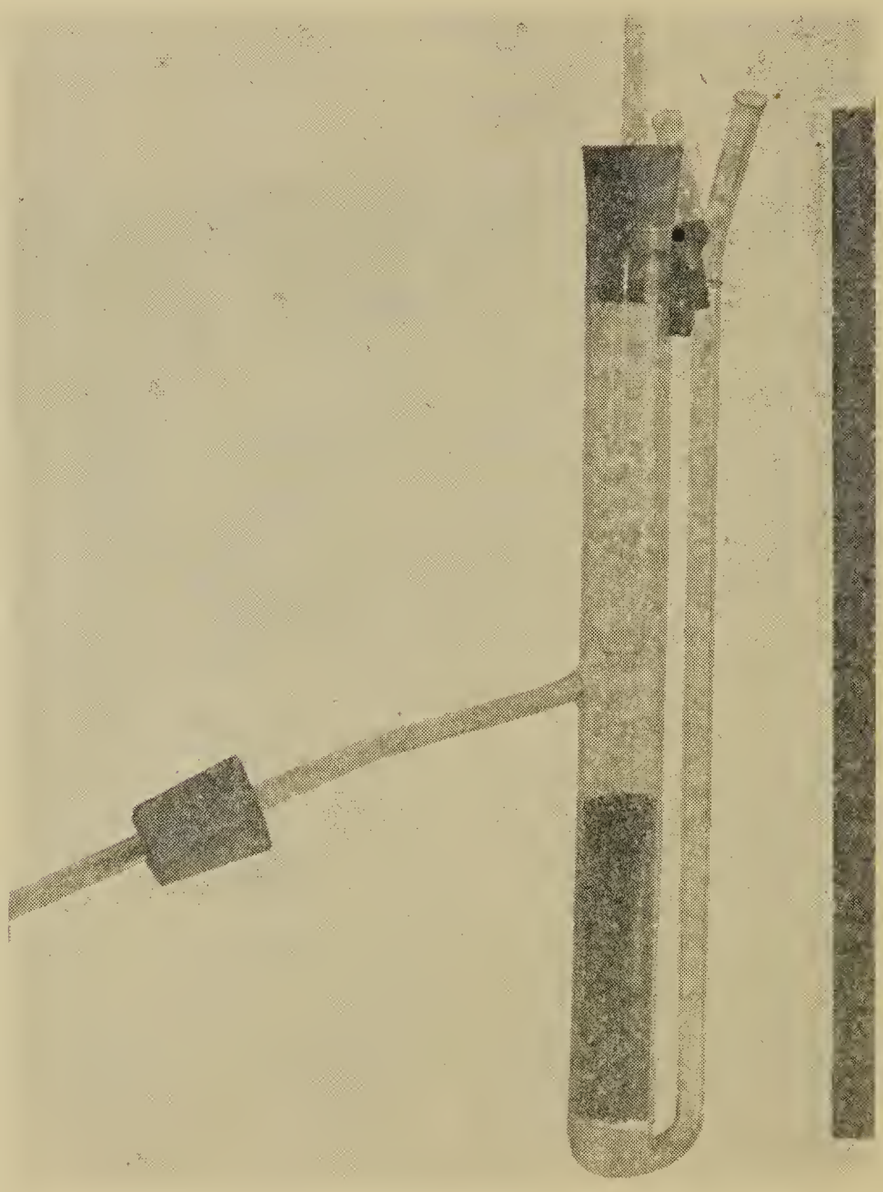

FIG. 6.

[For legend see end of paper.]

Chlorination of Methane.

Partial combustion problems by no means furnish the only examples of contact organic catalyses of marked exotherm. Of current interest because of the present large production of chlorine are chlorinations, such as the preparation of carbon tetrachloride from methane and free chlorine on a treated charcoal catalyst.

Taking the heat of this reaction as 83.14 kilogram calories and the molecular heat of chlorine as 7.8, and of methane as 9.6, it follows that the complete reaction with perfect heat insulation (assuming no decomposition and constant values for all of the quantities given) provides sufficient energy to raise the temperature of the incoming gas—

$$\frac{83{,}140}{4\,(7.8) + 9.6} = 2{,}038^\circ\ \text{C.}$$

Because of the assumptions made this figure is only illustrative, but when coupled with the known poor thermal conductivity of charcoal it shows at once that serious thermal problems must be solved before any such process can be put into large scale production. These problems are very materially increased by the severe limitations placed upon possible construction material by the very great chemical reactivity of chlorine at the favorable temperatures of reaction.

Possible Solutions.

The number of illustrations of contact catalyses of marked exotherm could be much extended, and there can be no doubt that the problems involved in the proper disposal of this exotherm are of much importance. Some of the possible methods of procedure are therefore of interest.

To absorb the heat of reaction it may in some cases be possible to use chemical means, that is, it may perhaps be possible to effect simultaneously an endothermic and an exothermic reaction; or to so change the reaction that it becomes endothermic. An interesting example of simultaneous exothermic and endothermic reactions (not, however, catalytic) is the simultaneous oxidation of carbon to CO and the reduction of carbon dioxide to CO by passing mixtures containing oxygen and carbon dioxide over heated carbon. The desired temperature can be controlled by altering the concentrations in the incoming gas mixture. This expedient was developed at Edgewood Arsenal and successfully used in the large scale production of CO for phosgene.[14] An example of apparently two different catalytic reactions yielding the same organic product from the same organic compound is the exothermic oxidation and endothermic dehydrogenation of methyl alcohol on copper to give

formaldehyde. Evidence presented by Le Blanc and Plaschke,[15] however, indicates that the primary reaction in both cases is dehydrogenation and that the valuable function of the oxygen is the maintenance of the dehydrogenating activity of the catalyst through alternate oxidations and reductions. A means whereby an active dehydrogenating catalyst might be maintained without permitting the simultaneous oxidation of hydrogen would certainly aid in the thermal problem here.

In certain cases it may be profitable to mount the catalyst directly upon the inside of the reaction tube and surround this with a liquid of high heat capacity. If necessary this liquid may be stirred. Since vapors at high temperatures and ordinary pressures occupy relatively large volumes per unit mass, the size of such an apparatus will probably confine the use of this expedient to reactions producing very valuable products.

A well-known expedient already referred to consists in increasing relatively the heat capacity of the incoming vapors by dilution with an inert material or an excess of one ingredient. This possesses the disadvantage that it greatly increases the volume of the incoming gases and so entails larger apparatus. Moreover, recovery difficulties are materially increased in those cases where the excess of inert material is gaseous and the product desired possesses an appreciable vapor tension. However, specific applications of the basic principle have been considered sufficiently valuable and novel enough to form the subject matter of recent patents.[16]

Another possibility is the utilization of the latent heat of vaporization of an atomized liquid carried into the reaction zone by the incoming gases. Credit for this suggestion is due to G. A. Hulett.

For large scale production the writer personally favors the principle of alternate coolings and short catalyst contacts. In this, the incoming gases are passed over a cooling coil immediately before they make contact with the catalyst. This cuts off possible preheating by radiation. The catalyst is mounted upon a cake or grid whose dimensions and temperature are so chosen that the reaction velocity and the time available for reaction shall permit only a fraction of that theoretically possible. This fraction is to be so chosen that the heat developed by the amount of reaction shall be insufficient to carry the temperature of the incoming cold

gases to the desired temperature, the excess being supplied by electrical heating from a resistance distributed uniformly throughout the grid, while the amount of current supplied is controlled automatically by an appropriate thermo-regulator. The hot gases then pass over a cooler capable of removing the heat developed, then over a second grid, and so on until the reaction is completed as desired. However, the practical application of this principle to reactions catalyzed only by non-metallic materials presents many difficulties which cannot be gone into here. Landis[17] claims to be able to maintain such a grid at uniform temperature in the ammonia oxidation, but from his patent specifications it is difficult to see how even he attains absolute temperature uniformity throughout the catalyst mass. At any rate, platinum gauze is much preferred to non-metallic catalyzers, and one of the points in its favor is the consequent simplification of the local overheating problem.[18]

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4. W. F. Rittman, C. B. Dutton and E. W. Dean—Manufacture of Gasoline and Benzene-Toluene from Petroleum and Other Hydrocarbons. Bureau of Mines Bulletin No. 114 (1916). Pages 43-45 give certain heats of reaction met with in the cracking of petroleum.

5. An apparatus for the investigation of some exothermic reactions in contact catalysis. The catalyst tube is surrounded by a molten salt mixture. Two thermocouple hot points, one imbedded in the center of the catalyst and one in the bath, give the temperature readings.

5*a*. Paul Sabatier—La Catalyse en Chimie Organique. Librairie Polytechnique, Ch. Beranger, Editeur. Paris (1913). Page 180, Tarry or carbonaceous matter on and molecular condensation in catalysts for the dehydration of alcohol.

5*b*. J. B. Senderens—Déshydration Catalytique des Alcools par Voie Sèche. (The Catalytic Dehydration of Alcohol by the Dry Process). Annales de Chimie et de Physique, Series 8, Vol. 25 (1912), 449-529. Chapter V, pp. 512-527, discusses the influence of heat upon the catalyzers.

6. Catalyst tube used in the apparatus shown in Fig. 5.

6*a*. H. D. Gibbs, et al.—Process of Oxidizing Aromatic Hydrocarbons having a Plurality of Side Chains, Canadian Patent 186,445, Sept. 10, 1918. Chemical Abstracts, Vol. 12 (1918), p. 2325. Hydrocarbons having an aromatic nucleus combined with either side chains or a ring at two points of the said nucleus, e. g., xylenes or anthracene, are oxidized by passing the hydrocarbon and an O-containing gas in contact with a vanadium oxide catalyst at 250°-650°C.

6*b*. Harry D. Gibbs and Courtney Conover—Process for the Manufacture of Phthalic Anhydrid, Phthalic Acid, Benzoic Acid, and Naphthaquinones. U. S. Patent No. 1,285,117. Patented Nov. 19, 1918. Vanadium oxide catalyst, 250°C. to 650°C.; preferred temperature 500°C. Also forms CO_2 and water.

6*c*. Harry D. Gibbs and Courtney Conover—Process for the Manufacture of Phthalic Anhydrid, Phthalic Acid, Benzoic Acid, and Naphthaquinones. U. S. Patent No. 1,284,888. Patented Nov. 12, 1918. Oxide of molybdenum catalyst and air, 250° to 650°C.; preferred temperature, 500° C. Also forms CO_2 and water.

6*d*. Selden Co. and H. D. Gibbs—Aldehydes, Carboxylic Acids, Phthalic Anhydrid; Quinones, Brit. 119,518, October 1, 1917. Chemical Abstracts, Vol. 13 (1919), p. 230. The oxidation of aromatic hydrocarbons containing a plurality of side chains, or a plurality of rings, or their lower oxidation products are affected by passing the vapor of the hydrocarbon mixed with air or other gas containing O, at 250-650° over a catalyst consisting of an oxide of a metal of the 5th or 6th periodic groups, *e. g.,* Sb, Bi, Cr, W, U, and especially V or Mo.

7. J. Walter—Einige Oxydationversuche durch theilweise Verbrennung. (A Research on Oxidation by Partial Combustion.) Journal für praktische Chemie, Series 2, Vol. 51 (1892), pp. 107-111.

8. F. G. Liljenroth—Starting and Stability Phenomena of Ammonia Oxidation and Similar Reactions. Chem. and Met. Eng., Vol. 19 (1918), pp. 287-293.

9. The Barrett Company—Oxidizing Benzene, British Patent No. 122,167, Nov. 12, 1918. Chemical Abstracts, Vol. 13 (1919), p. 1325, HCHO, Maleic Acid, etc., are obtained by passing benzene vapors mixed with O, air, etc., or gases containing O over a heated catalyst, such as V oxide.

10. Samuel Parsons Mulliken—A Method for the Identification of Pure Organic Compounds, Vol. 1 (1908), John Wiley and Sons, New York. Test 320, page 86 of Vol. I, Preparation of succin-*p*-toluide.

11*a*. William A. Bone and Richard V. Wheeler—The Slow Oxidation of Methane at Low Temperatures. Journal Chemical Society, London, Vol. 83 (1903), pp. 1074-1087. The oxidation of methane involves at least two distinct and successive changes, each of a simple character, namely, the simultaneous production of formaldehyde and steam, and the subsequent very rapid oxidation of the formaldehyde.

11*b*. William A. Bone and William E. Stockings—The Slow Combustion of Ethane. Journal of the Chemical Society of London, Vol. 85 (1904), pp. 693-727.

12. P. Sabatier and A. Mailhe—The Employment of Metallic Oxides as Oxidation Catalysts. Comp. rend., Vol. 142 (1906), pp. 1394-1395. Hydrocarbons of the methane series are destroyed almost completely to give carbon dioxide, water and aldehydes and acids of the methane series. CuO, NiO, CoO, etc., serve as catalysts. With O_2 reaction begins at 200°. Catalyst glows.

13. David Trevor Jones—The Thermal Decomposition of Hydrogenated Aromatic Hydrocarbons. Journal of the Chemical Society of London, Vol. 107 (1915), pp. 1582-1588. Cyclohexane, Me-cyclohexane, tetrahydronaphthalene begin to decompose between 490 and 510°—dihydronaphthalene begins at 390°. Cyclohexane gives benzene, acetylene (trace), ethylene, hydrogen, methane, ethane and higher olefines.

14. Col. William H. Walker—Gas, Smoke, and Flame in this War and the Next. Proceedings Engineers' Society Western Penna., Vol. 35 (1919), pp. 195-208. Page 198 describes the method of making CO. Photograph on page 213. Address, Tuesday evening, March 25, 1919. Metallurgical and Mining Section in joint meeting with the Pittsburgh Sections American Chemical Society and American Electrochemical Society.

15. M. Le Blanc and E. Plaschke—Uber die Darstellung von Formaldehyd aus Methylalkohol nach dem Kontaktverfahren. (The preparation of Formaldehyde from Methyl Alcohol by the Contact Process.) Zeit. f. Elektrochem., Vol. 17 (1911), pp. 45-57.

16*a*. Elektrizitätswerk Lonza—Alcohol from Acetaldehyde, Swiss Patent No. 77,471, April 16, 1918. Addition to 74,129, C. A. 11, p. 1884. Chemical Abstracts, Vol. 12 (1918), p. 2326. In the reduction of acetaldehyde by means of H in the presence of a catalyzer, as specified in the principal patent, the H is employed in such excess that the heat generated by the reaction is conducted away to such an extent that the temperature in the reaction chamber shall not exceed a definite predetermined point, i. e., 150°C.

16*b*. Elektrizitätswerk Lonza—Alcohol, British Patent No. 120,163, March 16, 1918. Chemical Abstracts, Vol. 13 (1919), p. 451. In the manufacture of alcohol by treating acetaldehyde vapor with H in the presence of a catalyst, the H is employed in a large excess over the theoretical quantity; this gives a product containing little aldehyde or undesirable by-products, and also serves to take up the heat developed in the reaction.

16*c*. T. Lichtenhahn—Ethyl Alcohol from Acetaldehyde, Canadian Patent No. 187,609, Nov. 26, 1918. Chemical Abstracts, Vol. 13 (1919), p. 134. EtOH is prepared from AcH by reduction with H in the presence of a catalyst, the H being used in such large excess that the heat of reaction of nearly 300 calories per kg. of EtOH produced is carried away by the H, so as to maintain a constant temperature favorable for a smooth reaction without decomposing the AcH.

17. Walter S. Landis—Catalyzer Apparatus for Oxidizing Ammonia. U. S. Patent No. 1,193,798, August 8, 1916. Describes precooler and grid catalyzer associated with powdered catalyzing material to be uniformly heated throughout its active mass, using electricity when necessary.

18. Charles L. Parsons—Commercial Oxidation of Ammonia to Nitric Acid. Jour. Ind. and Eng. Chem., Vol. 11 (1919), pp. 541-552. Page 544 gives advantages of platinum over non-metallic catalysts.

19. James F. Norris—The Manufacture of War Gases in Germany. Jour. Ind. and Eng. Chem., Vol. 11 (1919), pp. 817-828. Page 821 gives drawings and a brief description of the Badische plant for ethylene.

20. This impression was obtained at Colonel Walker's lecture in Pittsburgh on March 25, 1919, but does not appear in his published article. (Cf. Ref. No. 14.) Because of the value of the principle involved, mention of the expedient is made here with the reservation that the writer has not been able to confirm its use at Edgewood.

Chemical Research Laboratory,
Pittsburgh Experiment Station,
Bureau of Mines,
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THE THERMAL PROBLEM IN CONTACT SULPHURIC ACID MANUFACTURE.[1]

By F. C. Zeisberg.[2]

In discussing this subject it may be well, to begin with, briefly to outline what the sulphuric acid contact process is, and to review somewhat more fully the principles underlying it. It might be pointed out, also, that there are a number of different thermal problems in connection with the process considered as a whole—the one touched on in this paper is only that concerned in the conversion of SO_2 to SO_3.

As is well known, the essential feature of the contact process is the passage of a mixture of SO_2 and O_2 (air) over a suitable catalyst at a suitable temperature. The SO_2 is thereby converted to SO_3, according to the equation

$$2SO_2 + O_2 = 2SO_3 \qquad (1)$$

and 45.2 cal. are liberated. The gaseous SO_3 resulting is absorbed in water, employed usually in the form of weak sulphuric acid, thereby forming sulphuric acid.

The reaction shown in equation (1) is reversible and the equilibrium relationship among the various constituents of the system may be expressed by the equation

$$K = \frac{(SO_3)^2}{(SO_2)^2\ (O_2)} \qquad (2)$$

From this equation it is evident that an increase in concentration of the SO_3 in the mixture is brought about by an increase in either the SO_2 or O_2 concentration. Commercially, however, what is desired is not a maximum SO_3 concentration

[1] Manuscript received September 23, 1919.

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in the gases leaving the converter, but a maximum conversion of SO_2 to SO_3. This predicates the use of an excess of oxygen, and the reaction equation becomes

$$2SO_2 + nO_2 \leftrightarrows 2SO_3 + (n - 1)\ O_2 \qquad (3)$$

According to Knietsch n should be not less than 2, to get good commercial results.

If the source of SO_2 is the combustion of brimstone, as is the case in a good many commercial operations, the maximum amount of SO_2 which the burner gas may contain and not have less than two volumes of excess oxygen, is 7 percent, by volume. With a gas of this composition, *viz.*, 7 percent SO_2, 14 percent O_2 and 79 percent N_2, the maximum possible conversion may be calculated for a number of temperatures. Thus, for 400° C. the conversion is practically complete, *i. e.*, 100 percent; for 500° C. it is about 95 percent, for 600° about 80 percent, for 700° about 55 percent, and so on. In other words, it is evident that to obtain good conversions, and thereby good yields on the sulphur burned, it is necessary to carry on the conversion at temperatures certainly below 500° C. In fact, it may be asked, why not go to still lower temperatures, say 400° C., or even 300° C., and thereby make sure of 100 percent conversion.

The difficulty with this procedure is, however, that even when using platinum, the most active catalyst known for this reaction, the reaction velocity falls off so rapidly at the lower temperatures that except when employing gas flows much lower than are commercially allowable, sufficient time is not available, while the gas mixture is in contact with the catalyst, for equilibrium to be reached. In actual practice the conversion falls off even more rapidly per unit of temperature drop below 400° C., with the gas mixture in question, than it does per unit of temperature rise above 400°.

The upshot of these phenomena is that the sulphuric acid manufacturer is constrained to operate his converters within a rather narrow temperature range, say from 400° to 500° C., in order to obtain maximum conversion. And since the reaction velocity is rather low at the lower temperature the range actually becomes about 430° to 500° C., if his plant is being at all pushed for production.

It was remarked in the first part of this paper that 45.2 cal. are liberated when a molecule of SO_2 is converted to SO_3. This is heat sufficient to raise the temperature of the above gas approximately 400° C. if complete conversion is achieved. Since it is necessary to supply the gas to the converter at at least 400° C. in order to get the reaction to start, it is plainly evident that there must follow a sudden and uncontrollable rise in temperature, the maximum allowable limit for good conversion being quickly exceeded.

With an initial temperature of 400° C., the issuing gas would not rise to $400° + 400° = 800°$, as might at first glance be expected, because this temperature can be reached only if the SO_2 is completely converted to SO_3. It has already been seen that, as the temperature rises, the amount of SO_3 which may exist in equilibrium in the gas falls; at 800° C. less than 40 per cent of the SO_2 can be converted. This means that the maximum rise is less than 400°, a balance being struck when a temperature is reached at which no further SO_2 can be converted to SO_3. This rise is in practice, however, sufficiently great to constitute a real problem, the problem touched upon in the following.

In an exothermic, catalytic, gaseous reaction which must be carried out in a limited temperature range, because of some such condition as exists in this reaction under consideration, or for other reasons, such as the liability of the catalyst to deteriorate if overheated, there are several possible ways in which the temperature may be kept below the maximum limit.

1. The reacting gases may be supplied to the catalyst at so slow a rate that the heat is dissipated by natural, unassisted radiation.

2. The reacting gases may be diluted with some inert gas.

3. A portion of the converted gases may be returned and added to the incoming gas stream, thereby lowering the concentration of the reacting substances.

4. The conversion reaction may be carried out in steps, with intermediate cooling of the gases.

5. The bed of catalyst may contain cooling pipes and the converter be surrounded by a cooling jacket.

In order to understand clearly the present state of the art of contact sulphuric acid manufacture and its relation to the five possible methods of cooling mentioned above, it may be well to trace the development of the contact chambers as given in the American patent literature. There is appended a numbered list of U. S. patents, which is by no means complete, but certain examples of which will show the development clearly enough.

At first the contact chambers consisted of a set of parallel tubes, filled with platinized asbestos and surrounded by flue gases from a coal-fire. These tubes hence served as combined preheaters and converters. Since it was necessary to heat the tubes to near 400° C. to initiate the reaction, the centers of the tubes became very hot and as a consequence it was never possible to obtain good conversions. This type of converter manifestly did not embody any of the principles set forth above.

It was Rudolph Knietsch who first recognized the cause of the low conversion, resulting in his patenting (2) a multi-tubular converter in which the incoming gas passed up the outside of the tubes containing the platinized asbestos, being warmed by the heat of reaction radiated from the tubes. The warmed gas then passed down through the tubes. This converter was self-sustaining, no extraneous heat being applied to the gas. This type of converter employed the fifth principle, namely, the interlarding of the contact material with a cooling medium. It works fairly well, but the design is complicated and it is difficult to renew the contact mass. A whole series of patents has been granted to Knietsch on various modifications of his original converter, all, however, embodying the same principle of design, *viz.*, a group of parallel tubes.

The next development was made by Hasenbach (3) and (5) who employed the fourth principle. A preliminary conversion, running as high as 60 percent, was achieved by means of burnt pyrites, the resultant gas was cooled, purified, preheated and passed to a converter containing platinum, where an additional conversion of about 30 percent was obtained. This is the well-known Mannheim process which is still in use in this country.

About the same time Krauss and Wach (4) patented a converter in which an attempt was made to control the temperature

by using a series of catalyzer-carrying trays arranged in a tower. The preheated gas passed in at the bottom and where it issued from the first tray was mixed with a blast of cold burner gas introduced at that point. Cold gas could be introduced in regulated quantity between each pair of trays. The scheme does not seem practical, for wholly unconverted gas must always be introduced to cool the gas from the preceding tray, and the heat developed in converting this cold gas must always be more than enough to raise it beyond the temperature of the hot gas it is desired to cool.

The next attempt to convert in several stages was a patent to J. B. F. Herreshoff (13). Herreshoff uses two three-tray converter towers, arranged alternately in series with two heat exchangers wherein the exit gas gives up its heat to the entrance gas going to the first converter. This is a utilization of the fourth principle and is the forerunner of the process at present used by one of the foremost acid manufacturers of the country.

A rather novel adaptation of the first principle was covered in a patent (15) to W. C. Ferguson. He used three converters in series, supplying the first converter with a preheated burner gas containing about 11 percent SO_2. Only a partial conversion occurred, because of the high SO_2 concentration, and then additional cold air was introduced into the gas stream before it entered the second converter and the third converter. There is a small advantage in this method of dilution, over the method of originally diluting the burner gas to the same final degree, in that the heating of this diluting air to the temperature of initiation of the reaction is avoided. Nevertheless, as far as the writer is aware, this method is not in use in this country today.

Next appeared a series of patents to various individuals, covering various modifications of the fourth principle. Converters were made in sections, several separate converters were used with air cooling between them, and the principle of distributing the platinum concentration so as to regulate the amount of conversion occurring in the various portions of the converter or the different converters was brought forth. Even Knietsch, who had adhered until now to his multi-tubular condenser, patented (21) the separation of the mass in the tube into separately supported plugs, thus approximating the tray idea.

Eschellmann and Harmuth (Tentelev Process) attempted to transfer some of the heat of the reaction to the incoming gas by passing it down through a converter (22) which consisted first of a large empty chamber, on the bottom of which was disposed a layer of catalytic material, and then a set of parallel tubes containing catalyst. The layer of catalyst was supposed to radiate heat to the incoming gas. Since this gas is almost perfectly diathermous, however, it is difficult to see how it could be heated to any appreciable extent by this means.

Knietsch's next development was conversion in steps, each conversion being followed by a more or less complete absorption of the SO_3 formed. By thus removing the reaction product, more complete conversion with a smaller amount of catalyst was claimed. Since he employed heat exchange converters the cooling of the gas by the absorption of the SO_3 was not uneconomical. It is not believed this process was ever operated in the United States.

The more recent developments are the use of a multiple tray contact tower arranged so the sides can be insulated to any desired degree (W. C. Ferguson (25)) and various improvements in design of the apparatus for conversion in several steps, usually accompanied by heat interchange. Of this latter development the patent to T. Wolff (26) is representative.

In looking over this resumé it is evident that all of the five principles except the third have been used in the past. At present the principle most used is conversion in steps, usually in a tower-like converter containing several trays of catalyst alternating with large empty spaces in which the gas may mix before passing into the next catalyst layer. This empty space may contain cooling tubes, usually those of a heat-exchanger used to preheat the cold burner gas, or the walls thereof may be equipped to radiate heat. One large manufacturer uses the heat-exchanger principle, with the walls of the converter well insulated, another uses a converter the walls of which can be insulated to a variable degree, so that the excess heat is dissipated by radiation.

Either type of converter is quite satisfactory under present conditions. The arrangement of several trays affords the opportunity of varying the concentration and amount of catalyst in

each tray, by which means alone a considerable control over the heat development may be attained. The alternation of trays and mixing chambers permits an equalization of temperature which cannot help but be beneficial, for undoubtedly hot spots develop in each tray, due to easier gas passage at some points or accidental accumulation of excessive platinum in spots, and the gas coming from such a hot spot is overheated and not completely converted. By mixing with the larger body of cooler gas coming from the same tray, before passing on to the next tray, this overheating is corrected.

Some actual figures taken from a five-tray converter not fitted with a heat exchanger, which are representative of good operation are:

Entrance gas	385°C.
Exit from first tray	540°-560°C.
Exit from second tray	520°C.
Exit from third tray	500°C.
Exit from fourth tray	450°C.
Exit from fifth tray	385°C.

With these temperatures about 60 percent of the SO_2 is converted in the first section, the total conversion running a little over 96 percent.

While this style of converter, as has been mentioned above, is sufficiently satisfactory under present conditions, it is not at all certain to continue so. Very recently it has become evident that liquid SO_2 recovered from smelter fumes may be used as the source of sulphur in the contact process, instead of pyrites or brimstone. If this is done 9.5 percent of SO_2 may be present in the entrance gas, instead of the 7 percent when brimstone is burned, without having less than the 2 molecules of O_2 per molecule of SO_2 present which are recommended by Knietsch.

This richer entrance gas will result in higher temperatures in the converter, which may or may not be capable of being satisfactorily handled as at present. Consequently, though the problem of the disposal of heat in this particular reaction seems to have been fairly well solved at present, there is no certainty that it will remain solved and the chemical engineer will probably have ample opportunity to continue to exercise his ingenuity on this problem in the future.

REPRESENTATIVE U. S. CONVERTER PATENTS.

(1) 384,841—Haenisch and Schroeder.

(2) 652,119—Rudolph Knietsch—Method of Making Sulphuric Anhydrid.

(3) 670,559—Wilhelm Hasenbach—Apparatus for Making Sulphuric Anhydrid.

(4) 677,670—C. Krauss and J. Wach—Method of Making Sulphur Trioxide.

(5) 681,698—Wilhelm Hasenbach—Method of Making Sulphuric Anhydrid.

(6) 688,020—Rudolph Knietsch—Apparatus for the Manufacture of Sulphuric Anhydrid.

(7) 688,469—Rudolph Knietsch—Apparatus for the Manufacture of Sulphuric Anhydrid.

(8) 688,470—Rudolph Knietsch—Apparatus for the Manufacture of Sulphuric Anhydrid.

(9) 688,471—Rudolph Knietsch—Apparatus for the Manufacture of Sulphuric Anhydrid.

(10) 688,472—Rudolph Knietsch—Apparatus for the Manufacture of Sulphuric Anhydrid.

(11) 690,062—Rudolph Knietsch—Apparatus for the Manufacture of Sulphuric Anhydrid.

(12) 690,133—A. Clemm and W. Hasenbach—Process of Making Sulphuric Anhydrid.

(13) 719,332—J. B. F. Herreshoff—Method of Making Sulphuric Anhydrid.

(14) 719,333—J. B. F. Herreshoff—Apparatus for the Manufacture of Sulphuric Anhydrid.

(15) 723,595—W. C. Ferguson—Apparatus for Making Sulphuric Anhydrid.

(16) 723,596—W. C. Ferguson—Process of Making Sulphuric Anhydrid.

(17) 726,076—M. LeBlanc and C. Krauss—Process of Making Sulphuric Anhydrid.

(18) 731,758—C. Daub—Apparatus for Making Sulphuric Anhydrid.

(19) 736,876—E. Raynaud—Process of Making Sulphuric Anhydrid.

(20) 751,941—E. Raynaud—Process of Making Sulphuric Anhydrid.

(21) 774,083—R. Knietsch—Apparatus for Making Sulphuric Anhydrid.

(22) 792,205—G. Eschellmann and A. Harmuth—Catalytic Apparatus for Making Sulphuric Anhydrid.

(23) 809,450—R. Knietsch—Process of Making Sulphuric Anhydrid.

(24) 823,472—R. Knietsch—Process of Making Sulphuric Anhydrid.

(25) 857,389—W. C. Ferguson—Contact Chamber.

(26) 1,099,530—T. Wolff—Apparatus for the Chemical Transformation of Gases by Multiple-Contact Action at Regulable Temperatures.

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REACTIVITY AND ADSORPTION IN HETEROGENEOUS CATALYSIS.[1]

By Eric K. Rideal.[2]

During the last few years the importance and interesting behavior of heterogeneous catalytic reactions have attracted the attention of numerous investigators and have served to crystallize the various alternative theories, which have been propounded. The mechanism of heterogeneous catalysis naturally involves two separate investigations which at the present time have by no means been completed and may be summarized briefly in the two queries, firstly, in what way does the medium in juxtaposition to the catalyst differ from that in the free space? Secondly, by what mechanism is the reaction velocity increased or decreased by this alteration in the state of the medium?

The early work of Engler, Döbereiner, Wöhler and others on the influence of platinum on the combination of hydrogen and oxygen, of Deacon on the use of cuprous chloride in the hydrogen oxygen chlorine reactions laid stress upon the intermediate compound theory as a mode of operation. Faraday, on the other hand, was attracted by the remarkable increase in reaction velocity and by the influence of minute traces of poisons in catalytic reactions and stated quite definitely that in his opinion adsorption played a large part in such catalytic operations. He further hinted at the possibility of some other effect besides that of adsorption as being operative. The conception that adsorption is the primary action in all cases of heterogeneous catalysis is the viewpoint now generally adopted. It is also clearly recognized from such work as that of Firth on the adsorption of hydrogen by charcoal,[3] of Sieverts[4] on the adsorption of hydrogen by metals, especially

[1] Manuscript received September 26, 1919.
[2] Dept. of Physical Chemistry, University of Illinois.
[3] Zeit. Physik. Chem. (1914), 86, 299.
[4] Zeit. Physik. Chem. (1914), 88, 103, *et seq.*

palladium, and of McBain on the adsorption of iodine by charcoal[5] that the phenomenon of surface adsorption is frequently accompanied by an intra solid diffusion process resulting in the formation of solid solutions or compounds within the body of the solid material. Surface adsorption, it is stated, is an instantaneous reaction whilst intra solid diffusion is, as could be expected from analogy with Fick's law applied to liquid and gaseous systems, an extremely slow process. It is not readily evident that surface adsorption is instantaneous although it may be provisionally accepted to be so in the case of a plane clean metal surface exposed to a gas. Where, however, the solid consists of a honeycomb-like or fissured structure as in the case of active platinum black, desiccated colloidal oxides or even charcoal, the rate of transpiration of the gases through the pores and interstices of the material is by no means negligible. The work of Bredig (Anorganische Fermente) on the decomposition of hydrogen peroxide and some recent experiments by Greenwood, Taylor and the writer on the use of various support mediums for catalytic materials has however clearly indicated that the catalytic activity is proportional to the area of the catalytic material employed and consequently it follows that intra diffusion as well as transfusion of the reactants into the depth of the solid plays a small part in catalytic phenomena. Further work is still required to investigate exactly how thick a skin of solid is required to exert the maximum catalytic activity and again what relationship holds between the mean diameter of a capillary fissure and the depth to which the catalytically active surface will descend.

We cannot accept the conclusion that since Oberbeck[6] found that one molecular thickness of coherent metal was sufficient to alter the electrical potential of a platinum electrode, the same holds true for catalytic reactions; especially in the light of Pring's work on the extraordinary variation in the maximum thickness of different metals required to exert the maximum overpotential to hydrogen, a phenomenon in which catalysis probably plays a part. Evidence regarding the nature of the surface film which is instantaneously formed on the immersion of a solid in a liquid or gaseous medium and in which the catalytic reactions pre-

[5] Trans. Farad. Soc. (1919), **14**, 202.
[6] Wied. Ann. (1887), **31**, 337.

sumably take place is even less satisfactory than the general theory of adsorption. Faraday himself was of the opinion that it was several molecular thicknesses deep, an opinion strongly substantiated by the work of Bodenstein,[7] and especially Fink and Bodenstein, on heterogeneous catalytic reactions. Harkins and Langmuir, on the other hand,[8] postulate but a single layer. Lewis has shown[9] that Fink and Bodenstein's data on the catalytic oxidation of sulphur dioxide may be readily interpreted on either the diffusion or the single layer theory. In a recent paper the writer has shown that in gas mixtures containing carbon monoxide, hydrogen and oxygen undergoing surface combustion at the surface of various catalytic materials the ratio carbon monoxide to hydrogen burnt can be varied with the nature of the catalytic material. If diffusion processes alone governed the mechanism of catalysis it is difficult to understand how the ratio of the gases burnt should vary with catalytic materials. It was further found that at low temperatures carbon monoxide burnt much more rapidly than hydrogen, an unexpected result from the diffusion point of view, since we should expect that hydrogen with its greater mobility would penetrate most rapidly the adsorbed atmosphere of carbon dioxide and steam surrounding the catalytic material.

The very small influence of pressure on the rate of chemical combination indicates quite clearly that the pressure in the adsorbed film even if several molecules thick does not give an adequate explanation of the increase in the reaction velocity. It would appear that we must postulate some quasi chemical action between the reactants and the catalytic material, superimposed on, or distinct from adsorption phenomena. It is yet too early to state that powerful absorbing agents for reactants and products are good catalysts, although in many cases a general relationship appears to hold between the two phenomena. As an exception may be cited Patrick's silica gel, which, while possessing powerful absorbing qualities and possessing an immense superficial area (2,500,000 sq. cm. per gm.), is catalytically inert for most reactions. Two hypotheses have been advanced to explain why a quasi chemical combination between reactants and catalytic material accelerate a reaction, which proceeds but slowly when chemi-

[7] Zeit. Physik. Chem. (1903), etc.
[8] J. Amer. Chem. Soc. (1917), etc.
[9] A System of Physical Chemistry, vol. I, p. 472.

cal combination alone between the reactants occurs. The first assumes that the catalytic material exerts a directive molecular attraction which must vary in effective distance with the substance of the catalyst and of the reactants[10] and results in the adsorption of the reacting molecules in an orientated or polarized position.[11] It is then further assumed that neighboring molecules suitably orientated can rapidly combine with each other and the product of the reaction will more or less readily evaporate from the surface.

Langmuir cites several experiments conducted at low pressure in support of this hypothesis. Further support is given to this idea by the extraordinary variation in activity shown by different physical forms of the same catalytic material operating under the same conditions. A simple calculation will indicate how effective such a catalyst could be under ideal conditions. Taking Patrick's figure of 2.5×10^6 sq. cm. per gm. as the effective area of his silica gel, and a mean molecular diameter of 3×10^{-8} cm., the molecular effective surface area would be 2.5×10^{21} molecules per gm.; or 240 grams should be able to absorb a gram molecule of reactants ($N = 6.06 \times 10^{23}$). The effective surface in ordinary catalytic materials is far less than this, not only on account of the presence of poisons covering the surface but also because all the molecules on the surface of the material do not appear to exert this directive molecular attraction. The remarkable increase in activity exhibited by desiccated colloidal catalysts, by amorphous platinum and by the presence of small quantities of promoters all serve to indicate that molecules situated on the surface of materials may vary in activity, a phenomenon doubtless associated with the orientation of the fields of force or valency electrons around the atom. An alternative viewpoint has been developed by Lewis,[12] who, by combining the hypothesis of Marcelin and Rice,[13] that a molecule only reacts when its internal energy has been raised to a certain critical energy, and that of Trautz and Kruger[14] that the radiation (in these cases infra red radiation) is the source of energy which makes chemical reactivity possible, showed how to develop quantitative relationships

[10] Donnan, Phil. Mag. (1907), **1**, 647.
[11] J. Amer. Chem. Soc. (1917), etc.
[12] J. Chem. Soc. (1919), **115**, 182.
[13] Brit. Ass., p. 397 (1915).
[14] Zeit. Elektrochem. (1911), **17**, 453.

between the reactivity of the various molecular species in a reacting system and the radiation density in terms of the quantum theory. The reaction velocity of a reacting system is shown to be capable of formulation of the expression

$$\frac{dx}{dt} = \alpha \cdot e^{\frac{-E}{RT}}$$

Where α is a constant capable of evaluation and E the critical energy increment which must be contributed to a gram molecule of substance to bring it from the mean to the reactive state. Lewis assumes the validity of the quantum theory and equates $E = Nhv$ where h is Planck's constant and v the frequency of the absorbable radiant energy.

On the ordinary kinetic theory the number of molecules in a gram molecule which possesses a speed at any instant greater than v is given by the relationship

$$n = \frac{4N}{\alpha^3\sqrt{\pi}} \int_v^L e^{\frac{-\overline{v_0}^2}{\alpha^2}} \cdot v^2 dv$$

where α is an arbitrary constant and $\overline{v_0}$ the mean speed. It should thus be possible to develop from the known available data respecting the kinetic theory alone, similar relationships to those of Marcelin and Rice, without making direct use of the hypothesis of infra red radiation or the quantum theory, although there is much to be said in favor of these hypotheses.

In the application to heterogeneous catalysis, Lewis assumes that the sum of the critical energy increments necessary to activate the reactants at the surface of the catalytic material and the energy necessary to desorb the products of the reaction are less than those required to activate the reactants in the free state, and points out in support of the view that the thermal magnitudes involved in adsorption and desorption are relatively small.

These three theories may be roughly summarized as follows: On the view of Fink and Bodenstein, it would appear that every

molecule penetrating the diffusion layer to the catalyst surface reacts, the rate of catalysis being governed by the rate of diffusion of reactants or more usually of the products. On the single layer theory, only those molecules of the reactants undergo chemical combination which strike the catalyst in juxtaposition to one another, the rate of catalysis being usually governed by the rate of evaporation of the products from the surface. From the hypothesis of the critical energy increment we must assume that only those molecules in juxtaposition react when they attain a certain critical energy.

This hypothesis was applied by the writer to the example cited above, *viz.*, the simultaneous catalytic combustion of hydrogen and carbon monoxide. The relative rates of combustion of the two gases were calculated from the following expression

$$\frac{\frac{dx}{dt}\,\mathrm{CO}}{\frac{dx}{dt}\,\mathrm{H_2}} = \alpha e^{\frac{-E_{CO}+E_{H_2}+E_{H_2O}-E_{CO_2}}{RT}}$$

Where α, the ratio of the surface concentrations of the two gases, was assumed constant, E_{H_2O}, E_{CO_2} the energies of desorption of a gram mol. of the products of the combustion and taken equal to the latent heats at the temperature of operation, E_{H_2} the critical energy increment for the activation of hydrogen taken as 21,000 calories per gram mol. E_{CO} was calculated as follows: the equality of the four carbon valencies being assumed, since the heat of combustion of carbon to the monoxide is 26,100 calories and of the monoxide to the oxide 68,220 calories the heat equivalent of one carbon oxygen linkage is 21,060 calories. If we further assume that activation of the carbon monoxide requires the same critical energy increment as would be necessary to form carbon oxygen linkage we can take E_{CO} as 21,060 calories. In the following table are summarized the calculated figures for the ratio of carbon monoxide to hydrogen burnt on a catalytic surface and the actual ratios obtained utilizing oxide of iron as a catalytic material on a gas mixture containing 10 percent CO, 10 percent O_2 and 80 percent H_2.

Temperature ° C.	$CO:H_2$ Ratios	
	Calculated	Observed
400	1:8	1:9
375	1:7.2	1:7
350	1:6	1:6
325	1:5.2	1:5
300	1:4	1:3.2
275	1:1.5	1:1.5
250	1.5:1	5:1

Several interesting points will be noted from the above calculations, firstly that the critical increment of energy necessary to activate both hydrogen and carbon monoxide although calculated from entirely different bases are practically identical. This is in part confirmed by the fact that the ionizing potentials of these two gases lie within 0.2 volt of one another. It is well known that the line spectra of the elements are resolvable into definite series, which are taken to be representative of various oscillation frequencies of the valency electrons. The quantum theory gives us a method of estimating quantitatively the energy required of removing an electron from the atom or of forcing it into a new orbit; activation of a molecule therefore proceeds in stages and with definite energy increments corresponding with various oscillatory frequencies of the valency electrons until one or more of them finally leave the molecule or atom, when activation is complete. Again it will be noted that since $E_{CO} = E_{H_2}$ the ratio of the two gases burned depends entirely on the heat of desorption of the products CO_2 and H_2O. At, and above 400° C., the critical temperatures of both CO_2 and H_2O are passed and the theoretic ratio 1:8 depending only on the partial pressures of the two gases is reached. At temperatures below 400° and above 350° C. the critical temperature of CO_2 is exceeded, but not that of water. Thus the possibility of burning CO at a greater rate than hydrogen on a catalyst surface is attributable to the fact that water possesses a sensible latent heat of vaporization and the ratio of the two gases burnt is determined by the value of L_{H_2O} at the temperature of combustion.

From the figures cited above it will be noted that within the

temperature ranges 275° C. to 400° C. the agreement is excellent, but at lower temperatures it would appear that carbon monoxide is burnt too readily and is presumably preferentially absorbed or activated more easily than hydrogen. According to this viewpoint it is assumed that the ratios of the surface concentrations of each gas are independent of the catalytic material and thus the above ratios should hold for all materials provided that they are catalytic; experiments indicated that this was not the case since the ratio of $CO:H_2$ burnt varied with the catalytic material employed, as is indicated from the following figures:

Catalyst	$CO:H_2$ Combusted at 250° C.
Theoretical	1:0.20
Oxide of iron	1:0.75
Promoted oxide of iron	1:1.5
Oxide of copper	1:4
Nickel	1:5
Palladium on asbestos	1:8

It will be noted that there is a marked increase in the hydrogen undergoing combustion at the surface of catalytic materials which tend to adsorb hydrogen strongly, in other words the critical increment of energy necessary for activation of a reactant is not a unique property of the element but varies with the catalyst employed. It still remains to be determined whether such critical energy increments have any relationship to the calculated free energies of hypothetical compounds. It would appear, however, from a glance at the above table that we may range the critical energy increments with the free energies of the hydrides as far as they are known, or on the quantum theory forming a spectral series with them.

This observation naturally leads us to the viewpoint that if we assume activation of the gaseous molecules as part of the mechanism of catalysis, we should also imagine activation of the surface molecules of the catalytic material by the same means.

Adsorption compounds may therefore be regarded as two atoms becoming attached to the same electron, or the one electron traveling in an orbit common to both atoms, and to be distinguished from true chemical compounds where one electron has definitely left the sphere of influence of one atom to become forced in, or to oscillate around the other. The former is a compound on

Stark's hypothesis of atomic combination, the latter on Sir T. J. Thomson's.

Active catalysts should therefore be those in which the electron orbits are large (high atomic weight elements), possessing many small stages of activation (a dense line spectrum stretching far into the infra red) and should exert the maximum catalytic activity on those reactants which possess the same characteristics.

Mechanically we may regard the surface concentrations on strongly adsorbent surfaces as more than one molecule thick, due to virtual chemical action between the substrate and the first layer followed by adsorption on the second or third, on weakly adsorbing surfaces as only one molecule thick. On this hypothesis a catalytic material for the combination of hydrogen and oxygen at a definite temperature must possess the following characteristics, *viz.*, that the critical energy increment for the activation of hydrogen and oxygen at its surface must be less than that for the activation of hydrogen and oxygen in the free state (*e. g.*, Lewis gives a catalytic efficiency of 10^{17} for porcelain at 400° C.), and secondly the free energy of combination of hydrogen and oxygen in the free state at that temperature must exceed that of the reactants and the catalyst and desorption of the products of the reaction, *e. g.*, metallic sodium should not catalyze the above reaction owing to the formation of sodium oxide. Similar applications of the theory of activation and adsorption may likewise be made to heterogeneous catalytic reactions in liquid media, and it would appear possible in this case to express reactivities in units of electrical energy or since the Faraday is a universal constant in terms of electromotive force.

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DISCUSSION.

E. K. RIDEAL[1]: When I was on the other side of the Atlantic, I was put down by Dr. Taylor for a paper on Modern Theory of Matter and the Problem of Catalysis. We have devoted the whole of this morning to the problem of catalysis and apparently

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have come to no conclusion as to the mechanism of catalytic behavior; therefore I think it was extraordinarily unreasonable where we were so far apart, to put me down for two problems for which, as we know, there are no logical solutions at the present moment. But in excuse for the title, I would like to draw attention to one or two points which have been mentioned this morning but which appeared to me, possibly wrongly, to be of very great significance in the whole problem of catalysis. The first point I think we are all agreed upon, *viz.*, that the intermediate compound theory of heterogeneous catalysis has more or less gone by the board and we are confronted with the problem which is generally called adsorption, although various other names have been frequently popular in the scientific literature of the last four or five years. There are several investigations, especially those of Firth on Hydrogen, Sieverts on the Adsorption of Hydrogen by Metals, and McBane on Iodine and Charcoal; which show quite conclusively that there are two types of adsorption, a surface adsorption and diffusion, a slower process operating in the structure of the heterogeneous catalyst. It is generally assumed that surface adsorption is instantaneous or practically instantaneous. Dr. Fink told us that the SO_3-platinum reaction could not be measured by ordinary methods of determination and therefore we must regard this interdiffusion into the body of the catalytic material as playing no actual part in the catalytic operation itself. The work of Bredig on the Decomposition of Hydrogen Peroxide by Platinum, and Taylor and myself on Nitrogen and Hydrogen at high pressures, and also on the catalytic conversion of water gas at low temperatures, showed that the catalytic efficiency went up in proportion to the surface; therefore we must insist upon the point that it is the plain surface that is reacting. If we consider a uniform surface of metal, the problem arises "How deep can we deposit that metal on a support?" With any type of inert support we can mention, such as pumice and various forms of silica, we must inquire "How thick must the metal be before the catalytic efficiency rises to a maximum?" Pring, many years ago, showed that in electro-potential measurements one molecule thick was sufficient to alter the character of the electrode, one molecule of platinum on the surface of copper was sufficient to turn the copper electrode into a platinum elec-

trode. Is that true of catalysis? Personally I do not know; I think it is.

COLIN G. FINK[2]: Mr. Rideal referred to the film thickness of the adsorbed final compound. I really do not see the necessity of having several theories as to the film thickness. Furthermore, there is no necessity of relying so much upon hypothesis when, as a matter of fact, you can measure the film thickness. In the case of SO_3 this was readily determined.[3] I found by carefully measuring the quantity of SO_3 and by determining the extent of the surface that the film thickness corresponded to one molecule; not several, but one molecule. Now the question arose at once that a platinum surface which appears perfectly polished and smooth to the naked eye is, upon magnification, a very rough surface. Accordingly, on the basis of the assumption that it was a perfectly smooth surface, the molecular thickness was equal to one; however, on the assumption that the surface is rough, the thickness becomes less than one.

E. K. RIDEAL: I might just make a point there concerning this idea of film thickness. Of course, with strongly adsorbent substances it is evident that we can imagine that if the electron is moved as we expect, in the case of palladium and hydrogen, very close to the palladium, that we ought to be able to even put another hydrogen atom on top of this adsorbed hydrogen, and this virtually becomes our catalytic surface. I should be quite ready to admit, with Dr. Fink. that the film thickness need not necessarily be one or even two molecules thick as it would be quite possible to build up an orientated chain depending, naturally, upon the increased activation of the gaseous hydrogen to make it react with the new surface layer, which is now less reactive than the palladium was before. Furthermore, we ought to be able to calculate the energy of activation of the first hydrogen, the second, third, etc. We shall find that necessarily the activation increases. Finally the point will come when the activation necessary will become so great that the reaction will go on by itself in the gaseous medium and it will no longer be necessary for the gas to be adsorbed, *i. e.*, surface catalysis will cease. But in the case of strongly adsorbent substances, like palladium for hydrogen

[2] Head of Research Laboratories, Chile Exploration Co., New York City.
[3] Zeit. Phys. Chem. (1907), 60, 1-55.

and sodium for oxygen, it is evident that the cases are somewhat different. In the second case we must imagine that the atom of oxygen has moved so far into the sphere of the sodium that it is a chemical compound, *i. e.*, it is not dissociated again within the ordinary temperature ranges of catalytic operations.

COLIN G. FINK: I should like to refer to a very simple method of Bodenstein's of controlling the film thickness in the case of a metal catalyzer, in particular. You build your catalyzer, say platinum for example, or palladium, in the shape of a small bulb, and then place your glass vessel around that; then you can run a cooling or a heating solution through your bulb, and, say in the case of detonating gas, the hydrogen plus oxygen reaction, which Bodenstein studied, shows that the thickness of the film can be regulated by the temperature of your catalyst, which would either make it infinitely thick, so that you could measure it in microns, or you could make it very thin. We always found that no matter how high we got the temperature of the platinum, that there was a m i n i m u m thickness of the film of H_2O below which we could not drop. I do not believe you can get below one molecule thickness. However, the a v e r a g e thickness may be less than one molecule if your catalytic surface is not equally active over the entire extent. This seems more logical than assuming actual thicknesses of only half a molecule or a third of a molecule. The tenacity of the film seems to me to imply continuity and unimolecular thickness (not less).

Discussion in Connection with the Experimental Demonstration of the Preparation of Fluorine, offered at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 26, 1919.

DEMONSTRATION OF PREPARATION OF FLUORINE.

By F. C. Mathers.

F. C. Mathers[1]: You are familiar with the wonderful classical experiment of Moissan in which he prepared fluorine by the electrolysis of a solution of potassium hydrogen fluoride in anhydrous hydrofluoric acid, at a low temperature. The difficulty of making anhydrous hydrofluoric acid can only be appreciated by those who have tried to do it. Since the anhydrous acid is more corrosive and active than ordinary aqueous hydrofluoric acid and since it boils at 19° C., the difficulties in preparing and in using it in the Moissan apparatus are very great. It is necessary to cool the electrolytic apparatus with solid carbon dioxide and ether. These difficulties, while they cannot be avoided, are not impossible, but the very rapid corrosion or solution of the platinum anodes during the electrolysis is an unavoidable and very serious difficulty. Many of the descriptions of the Moissan apparatus do not especially mention this fact. He states that he used club-shaped anodes because they lasted longer. This corrosion of the platinum anode was so very serious that we were not very successful with his apparatus and method. So far as I know, fluorine has not been made in this country except in one or two instances by this process. I know of one place where rather carefully tried experiments failed. It has been made by this Moissan method in many different researches by O. Ruff in the Technischen Hochschule, Breslau, and he has made quite a reputation[2] from these experiments. He makes the statement that the preparation of fluorine by this method will always be a very difficult laboratory manipulation on account of the trouble in making, keeping and using the anhydrous hydrofluoric acid.

[1] University of Indiana, Bloomington.

[2] Die Chemie des Fluors, von Dr. Otto Ruff. Julius Springer, Berlin, 1920.

In the apparatus and method shown here, many of the above inherent difficulties encountered in the Moissan method have been avoided.[3] First the electrolyte is melted potassium hydrogen fluoride, whereby the easily maintained temperature of 225° C. is substituted for the very difficultly obtained temperature of —40° C. in the Moissan method. Second: the easily prepared potassium hydrogen fluoride, which is our electrolyte, is merely a starting material for making the anhydrous hydrofluoric acid required for the Moissan method. Third: graphite can be used as anode in place of platinum. I may add that it is impossible to use graphite in the Moissan method—it disintegrates in a few minutes. Platinum cannot be used in the fused potassium hydrogen fluoride electrolyte: it dissolves readily. Fourth: there are no great difficulties in making the apparatus for the fused bath process. The entire apparatus can be made of copper with the joints brazed with silver solder. I have here the diaphragm of the original apparatus with which we first made fluorine. It is simply a funnel-shaped affair fashioned from sheet copper with a copper outlet-tube leading from the small end. The joints and seams were brazed with silver solder. A piece of graphite, for anode, was fastened with a copper wire underneath the diaphragm. You see the diaphragm was always electrically connected to the anode, but the copper is resistant enough to corrosion so that considerable fluorine was made before this hole was eaten in the side of the diaphragm.

It is best to insulate the anode from the diaphragm. The copper connection to the graphite anode inside the diaphragm should pass a stuffing box soldered to the top of the diaphragm. This stuffing box, with the anode wire passing through it, is filled full and rammed tightly with moistened powdered fluorspar. The stuffing box can be filled with melted potassium hydrogen fluoride which is satisfactory as long as the temperature of the top of the diaphragm is kept below the melting point of that salt. Copper was the most resistant of all the metals tried. Platinum dissolves very rapidly, and stellite, nichrome, silver, gold are all readily attacked or dissolved. Graphite is a better material than copper, in that it is free from attack. The only serious difficulty with it

[3] Trans. Amer. Electrochem. Soc. (1919), **35**, 335.
Jour. Phys. Chem. (1919), **23**, 348.

is that it occasionally has faulty or porous places where the molten electrolyte leaks through. We try to use sufficiently thick walls so that no faults will reach entirely through. The diaphragm in this apparatus, being demonstrated, has been cut from a solid piece of Acheson graphite. The hole through the center of the top, through which the anode connection is made, is filled with molten potassium hydrogen fluoride for insulation. The bath into which the diaphragm dips contains about 2.5 pounds (1.25 kilos) of potassium hydrogen fluoride. Graphite is the best material for this vessel, although copper can be used. A separate current passing through heating coils is used to melt the bath. I shall not be able to show all of the experiments that I intended because I have been unable to secure the proper resistance. The current I am using is too small to give the volume of fluorine needed. Those persons close enough to the apparatus can notice the peculiar odor of fluorine. It is something like chlorine but much less irritating and has the suggestion of ozone. The sharp irritating odor is due to hydrofluoric acid, which is constantly being lost from the bath. This loss is so great that we only get something like a 40 percent material yield counting from the hydrofluoric acid. The free fluorine is not poisonous, unfortunately, since the idea in these experiments was to produce a toxic substance for chemical warfare work. With no protection whatever we have worked in a noticeable concentration of it for several weeks without ill effects. Its outstanding property is its extreme chemical activity. It combines with organic substances with sufficient vigor to start combustion. Sulphur, wood, charcoal, filter paper, cotton, alcohol, or other organic liquid introduced on a stirring rod, combustible gases, tin foil, etc., all burst into flame when held in the stream of fluorine gas coming from the apparatus. I shall only be able to show the more vigorous of these reactions. Flowers of sulphur instantly take fire. A wood splint first chars and then ignites.

I feel that this process opens up the possibility of making fluorine in considerable quantity in any laboratory. There are really no special difficulties in the manipulation of the apparatus. We were easily able to change 100 gm. of selenium into the fluoride in an afternoon, with a cruder, smaller apparatus than this one. The importance of this apparatus and method for re-

search work is apparent when we know that Ruff only prepared 15 to 20 gm. of the final fluorides in some of his researches. You see the great field for research work with this apparatus, whereby large quantities of fluorine are readily made available.

I should mention one thing: potassium hydrogen fluoride when freshly prepared, melted and electrolyzed, will not give fluorine. A number of hours of heating and electrolysis are required before any fluorine is obtained. This particular bath was heated and electrolyzed for nine hours before I obtained fluorine. It is hard to believe that water stays in the molten bath for that length of time, but I have no other explanation. After a bath has once been heated until it gives fluorine, it may be kept any length of time and will immediately give fluorine as soon as it is melted again and electrolyzed. When not being used the cell should be protected from moisture by storage in a large can having a tightly fitting cover. I am emphasizing the necessity of the dehydration of the electrolyte in order that none of you may become discouraged and condemn the method if you do not obtain fluorine immediately in your experiments. The real difficulty with this process is the impossibility of regenerating the bath in the cell itself during electrolysis. The operation cannot be made continuous. The electrolysis used up hydrofluoric acid from the double salt, hence the melting point of the bath increases. If a higher heat is used in order to maintain a fluid condition, hydrofluoric acid is more rapidly driven from the cell. Regeneration can be accomplished by pouring the electrolyte from the cell at the end of a run, grinding, treating with sufficient aqueous hydrofluoric acid, remelting and dehydrating. We were unable to find any method of regenerating by passing hydrofluoric acid gas into the molten bath.

This particular cell which I am showing was made entirely by Mr. Humiston, one of the other chemists who worked upon this research.

COLIN G. FINK[4]: May I ask Mr. Mathers, is there any chance of a commercial utilization of fluorine on a large scale?

F. C. MATHERS: I do not know of any possibilities. A patent is being taken out on the process and the apparatus, through the

[4] Head of Research Laboratory, Chile Exploration Co., New York City.

War Department. It looks to me as if it simply opens up the possibility for some valuable research work in university laboratories.

S. A. TUCKER[5]: How do you handle it for those reactions? How do you hold it?

F. C. MATHERS: We handle it through copper; we were able to get copper tubing of various sizes and connected the copper tubing with small brass fittings. Zinc is soon eaten out of the surface of the brass, but they hold very well. We tried soldering the copper together, but the solder was eaten up in the course of a little while, and we had trouble. The only thing we found from which we could make a permanent joint was silver solder; that holds very well; it is not eaten up rapidly.

S. A. TUCKER: What does it do to glass?

F. C. MATHERS: As a matter of fact we are not able to prepare any of the samples of fluorides and keep them in glass. We made a number of the various fluorides but had trouble keeping them. If we put selenium fluoride in a copper vessel, it reacted with the copper, and if we put it in paraffine, it reacted with the paraffine. Graphite was too porous. After making these things there is difficulty in keeping and using them. What we needed last year was platinum apparatus; we should have had platinum bottles for holding these samples; Ruff had apparently a wonderful outfit of platinum.

W. D. BANCROFT[6]: You can keep it for a moderate length of time in a glass tube?

F. C. MATHERS: Yes, for a few hours, but a gradual change was readily noticeable.

S. A. TUCKER: Concerning having to heat the electrolyte before you get fluorine, in the preparation of metallic calcium, you have similar phenomena. I think it is a matter of electrolysis, to a large extent.

F. C. MATHERS: Well, we both heat it and electrolyze it, but if we heat it long enough it will give the fluorine; in other words, it will be dehydrated.

[5] Columbia University, New York.
[6] Professor of Physical Chemistry, Cornell University.

S. A. TUCKER: There is an experiment I asked Col. Bancroft about in Washington, and that is the possibility of seeing what would happen by the interaction of argon and fluorine; there you have two elements having peculiar properties and might possibly get a combination for the first time.

MR. LYNN: Have you tried any with liquid air?

F. C. MATHERS: Not with liquid air. The lowest cooling we had was with carbon dioxide and ether. I might say that the literature on these various fluorides is apparently very inaccurate. We had indications that there were two carbon fluorides formed and two selenium fluorides and sulphur fluorides; there seems to be no question but that those are produced, but the literature says very little about it. There has not been much investigation on these compounds, owing to the difficulty of making the fluorine.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

FACTORS GOVERNING THE STRUCTURE OF ELECTRODEPOSITED METALS.*

By William Blum.[1]

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1. INTRODUCTION.

In spite of the great number and variety of papers published upon electrodeposition it is significant that few attempts at generalization have been made. In consequence we find even in the results of scientific research a tendency toward the recommendation of empirical formulas and conditions which are not correlated and sometimes contradictory. If rapid progress is to be made by investigations in this field it is essential that such studies should be conducted from the standpoint of general principles, so that the results obtained in any given study, may be applicable, in some degree at least, to other kinds and methods of plating. Only by some such course may the electrochemists of this country render adequate service to the electroplaters in their admittedly empirical art.

* Manuscript received July 31, 1919.

[1] Published by permission of the Director of the Bureau of Standards.

Of the suggested generalizations in this field the most comprehensive are those contained in Bancroft's so-called "axioms of electroplating" first offered[2] in 1904 and reiterated in 1913.[3] On the latter occasion Prof. Bancroft invited discussion and criticism of these axioms, but so far as known to the author no systematic experiments have been conducted to determine their validity since the original experiments of Bancroft and Snowdon. In an extended study at this Bureau of deposits from the silver voltameter[4] the results obtained were in accord with the above-mentioned "axioms." In this latter paper an explanation is offered for the effects of current density, temperature, concentration, and certain impurities.

One reason for the lack of discussion or application of these axioms by electroplaters is the fact, pointed out by Mr. G. B. Hogaboom in 1913, that few platers could even understand, much less employ them. In order, therefore, to translate these axioms into platers' language and to illustrate their application to electroplating, a number of simple experiments were recently conducted at the Bureau of Standards, the results of which were presented in an informal report to the American Electroplaters' Society at their annual convention in Philadelphia in July, 1919. Since these experiments were conducted as illustrations rather than demonstrations of the various effects, no attempt has been made to refer in detail to the voluminous literature upon the subject. Even though the experiments were primarily pedagogical and in no sense conclusive or exhaustive, it is believed that the results may be of some interest to the Electrochemical Society and may serve to invite discussion of these axioms or other principles of electrodeposition. If some such principles can be definitely established and accepted as a working basis for research in this field, an economy of time and effort will be effected for both the investigator and the commercial operator.

2. PRINCIPLES INVOLVED.

These experiments were largely based upon Prof. Bancroft's axioms, which were stated by him in the following form:

[2] Trans. Amer. Electrochem. Soc. (1904), 6, 27.
Jour. Phys. Chem. (1905), 9, 277.
[3] Trans. Amer. Electrochem. Soc. (1913), 23, 266.
[4] E. B. Rosa, G. W. Vinal and A. S. McDaniel, Bureau of Standards, Reprint 195 (1912).

1. Bad deposits are due to excessive admixture of some compound or to excessively large crystals.

2. Excessive admixture of any compound can be eliminated by changing the conditions so that the compound cannot precipitate.

3. Increasing the current density, increasing the potential difference at the cathode, or lowering the temperature, decreases the size of the crystals.

4. The crystal size is decreased when there are present, at the cathode surface, substances which are adsorbed by the deposited metal.

5. If a given solution will give a good deposit at any current density, it will give a good deposit at any higher current density, provided the conditions at the cathode surface are kept constant.

6. Treeing is facilitated by a high potential drop through the solution and by conditions favorable to the formation of large crystals.

It should be noted that these axioms are confined to the effect of certain conditions or variables upon the structure of the deposit and have no direct or necessary relation to the purity of the metal obtained or to the electrode efficiencies or to numerous other factors which may play a very vital part in the success of a plating operation. They are however of special interest to the electroplaters, who are concerned principally in the appearance rather than the composition of the finished deposits. From the point of view of the plater we may usually define a "good deposit" as one which is fine grained, since fineness of grain is in general accompanied by a high lustre, relative freedom from porosity and comparative hardness. In electrotyping, the structure of the metal may be even more important since it largely determines the physical properties upon which the durability of the product depends. It is therefore highly desirable to define, if possible, conditions of operation which will produce a fine structure (or which in some cases will permit any change in structure required to produce a desired finish), which conditions should be susceptible of control and should permit favorable operation over a considerable range.

In considering the structure of the deposits it is desirable to

distinguish between the size and shape of the actual crystals, and the structure of the deposit as a whole. The latter may be influenced by a number of factors, other than crystal size, which must be included in any comprehensive study of the structure of deposits. In the experiments to be described no attempt was made to determine the causes of pits, ridges, cracking, peeling or numerous other defects which may be encountered in plating, and which are probably due to specific properties of the particular metal or its salts and hence are less readily subject to generalization.

3. OUTLINE OF EXPERIMENTS.

In order to obtain information as to the applicability of the proposed axioms, simple experiments were conducted with solutions of nickel, zinc, and copper, in which the purpose was not so much to obtain good deposits from the platers' standpoint, as to furnish illustrations of the effect of a given variable. In order, therefore, to make the effect of the particular variable pronounced, it was often fixed at a value known to be outside of present good working practice. The effort was made to control conditions so as to confine the effect to one variable. This was not always feasible since obviously it is not possible, *e. g.*, to change the conductivity of a solution without changing some other properties. It is believed, however, that qualitatively the effects noted were due principally to the factor designated.

It was found most satisfactory to define the concentrations in terms of normality (except for substances such as glue of indefinite molecular weight or constitution). In view of the obvious advantage of this practice in chemical work it would hardly deserve mention were it not for the custom in such investigations (including some previously published by the author) to fix the concentration in grams per liter, selected either arbitrarily as *e. g.* 200 g/L or with a view to ready conversion to ounces per gallon, thus 45 g/L (6 oz./gal.); or even to use the indefinite term percent. The use of "grams per liter" or "ounces per gallon" is fully justified in expressing the results of an analysis or the formula to be used in preparing a solution, but in conducting experiments the use of normalities is preferable, since it permits a ready conception of the "constitution" and probable behavior of the solution

and the rôle played by any addition. Misconceptions may arise from failure to compute the normalities, especially when substances of low equivalent weight are added to solutions containing metals of high equivalent weight. Thus, if a solution is defined as containing 10 percent of lead acetate and 5 percent of nitric acid, or 100 g/L of lead acetate and 50 g/L of nitric acid, it would appear to consist principally of lead acetate. If, however, the same solution is defined as 0.45 *N* in lead acetate and 0.79 *N* in nitric acid, it is apparent that the solution may with even greater accuracy be considered as consisting of lead nitrate with the addition of acetic and nitric acids. In a few cases where there may be uncertainty as to the equivalent weight, as *e. g.* with organic compounds, confusion may be avoided by stating the molarity rather than the normality.

In order to avoid the introduction of impurities, the purest commercial rolled metals were used as the anodes. Owing to the passivity of pure nickel it was necessary to add nickel chloride to the nickel baths to produce adequate anode corrosion and thus avoid changes in concentration or acidity. In all of the baths a definite appreciable acidity was maintained in order that slight differences in anode and cathode efficiency would not cause appreciable changes in acidity during a run. This was accomplished by the presence of boric acid in the nickel and zinc solutions and of dilute (0.1 *N*) sulphuric acid in the copper solutions.

In order to avoid any deposition by immersion with its attendant possible effects upon the structure of the deposits, the latter were prepared in each case upon the corresponding metal, *e. g.*, nickel upon nickel, etc. The cathodes were all rolled metal, fairly smooth and uniform, so that the deposits obtained in any case are comparable with other deposits of the same metal. Except for cases that will be cited, all deposits were made of the same thickness (0.025 mm. or 0.001 inch) since otherwise the size of the crystals would not be comparable. This thickness was selected as representing a fair average for most electroplating. More pronounced differences would no doubt be obtained with thicker deposits, especially if cross sections were to be studied. The details and results of the various experiments will be considered from the standpoint of operating factors rather than in the order suggested by Prof. Bancroft.

4. RESULTS OF EXPERIMENTS.

A. Current Density, Concentration and Agitation.

In discussing the effect of current density it is convenient to combine axiom 5 with part of axiom 3 in the following statement: "Increasing the current density decreases the size of the crystals. If a given solution will give a good deposit at any current density it will give a good deposit at any higher current density, provided the conditions at the cathode are kept constant." Since the current density determines the rate of deposition and consequently the output of a given plant, great stress is usually laid by the platers upon "rapid solutions," *i. e.,* those which will permit high current densities. To many platers the first part of the above statement appears incorrect since it is well known that upon increasing the current density above a certain value the deposits become coarse and spongy with the characteristic "burnt" appearance. This effect, usually due to the co-precipitation of hydrogen, may properly be considered as an illustration of Bancroft's first axiom, that "bad deposits are due to excessive admixture of some compound," provided that in it we substitute "substance" for "compound." In order to illustrate the effect of increasing the current density under normal conditions, deposits were produced at current densities from 0.2 to 3 amp./sq. dm. (1.9 to 28 amp./sq. ft.), in the following solutions, which served as "standard" solutions for purposes of comparison. These solutions are in no sense suggested as commercial standard plating solutions, but were adopted merely as simple solutions from which good typical deposits could be obtained.

(1) Copper sulphate *N* (125 g/L or 17 oz/gal)
Sulphuric acid 0.1 *N* (4.9 g/L or 0.7 oz/gal)

(2) Zinc sulphate *N* (144 g/L or 19 oz/gal)
Boric acid 0.1 *M* (6.2 g/L or 0.8 oz/gal)

(3) Nickel sulphate 0.8 *N* (112 g/L or 15 oz/gal)
Nickel chloride 0.2 *N* (24 g/L or 3.2 oz/gal)
Boric acid 0.1 *M* (6.2 g/L or 0.8 oz/gal)

Photographs of the deposits with a magnification of 20 diameters are shown in Figs. 1 to 3.

From Fig. 1 it may be seen that with copper the effect of increasing the current density from 0.2 to 2 amp./sq. dm. is to produce a marked decrease in the size of the crystals. A further

increase to 3 amp./sq. dm. produced no appreciable change in the crystal size, but tended to produce a porous or "burnt" deposit. The actual difference in the size of crystals produced with different current densities is emphasized in Fig. 4, which represents the cross section (300 diameters) of heavier deposits of copper produced in agitated electrotyping baths in connection with a previous investigation[5] at this bureau.

Fig. 2 shows that with zinc also the effect of increased current density is to diminish the size of the crystals. The roughness of the first two specimens is characteristic of zinc deposits produced from sulphate solutions at relatively low current densities, and is probably connected with the fact that under such conditions bubbles of hydrogen are liberated slowly and cling tenaciously to the zinc surface.

Fig. 3 shows with nickel the same beneficial effect of increased current density, exemplified by the deposit at 2 amp./sq. dm., which is very bright and almost non-crystalline. The characteristic curling and cracking of nickel at higher current densities is no doubt due to the increased occlusion of hydrogen.

Having shown that under any given conditions, an increase in current density up to a certain point decreases the size of the crystals and therefore (for most purposes) improves the deposit, it is important to determine by what means this critical current density can be increased. Since this point is probably that at which hydrogen evolution becomes excessive, because of local impoverishment of the solution, it is obvious that either an actual or a local increase in metal concentration will reduce the tendency toward spongy deposits; in other words, the conditions at the cathode will be kept more nearly constant. The simplest way to maintain the metal concentration at the cathode is by means of agitation such as is produced commercially by compressed air or by mechanical means. If the result of such agitation is to increase the effective concentration of the metal, its influence (at any given current density) should be much more marked in a dilute than in a concentrated solution.

To test and illustrate this point, deposits were produced in solutions which were respectively twice, and one-half the con-

[5] W. Blum, H. D. Holler and H. S. Rawdon: Trans. Amer. Electrochem. Soc. (1916), 30, 53-66.

centration previously employed, at a current density of 1 amp./sq. dm. (9.3 amp./sq. ft.), in (a) still solutions, (b) those agitated with compressed air, and (c) those with mechanical agitation produced by rotating a glass stirrer between the anode and cathode. The results obtained are illustrated in Figs. 5 to 7.

From these results it may be seen that with all three metals agitation of dilute solutions produces much smoother deposits than are obtained in still solutions, while with the more concentrated solutions (at the current density employed) agitation produces no marked improvement except in the case of zinc. Here the removal of the ridges by the agitation is probably due to the sweeping away of bubbles of hydrogen. No consistent difference was observed between the effects of air and mechanical agitation. In some solutions, however, the use of air for this purpose may be objectionable, as was found to be the case in lead fluoborate solutions. In view of the marked advantages of agitation in permitting higher current densities, especially with dilute solutions, it at first appears surprising that so much plating is conducted in still solutions. One objection to agitation is the stirring up of sediment, which causes roughness. In barrel plating this effect is counteracted by the burnishing action produced by the agitation. In many mechanical plating devices a fair degree of agitation is secured without stirring up the sediment. A more general application of agitation in commercial plating seems highly desirable.

B. Temperature.

In order to determine the effect of temperature upon the structure, deposits were produced in the original ("standard") solutions at a current density of 1 amp./sq. dm. (9.3 amp./sq. ft.) at 25° C. (77° F.) and 65° C. (149° F.).

The results, as shown in Figs. 8, 9 and 10, confirm in every case the statement of Prof. Bancroft, that "lowering the temperature decreases the size of the crystals." In every case the deposits at higher temperatures show a coarser crystalline structure, least marked with nickel. The difference is even more noticeable in Fig. 11, showing cross sections of copper produced in electrotyping baths over an even smaller range in temperature. This conclusion seems at first to be at variance with the experience of

platers, many of whom employ hot solutions to obtain smoother deposits. Where hot solutions are advantageous it is probably due to other causes such as (a) an increase in the solubility of the salts, (b) an increase in the conductivity (and consequent decrease in the tendency to treeing), or (c) a decrease in the occlusion of hydrogen by the deposit (*e. g.,* with nickel). This is a good illustration of the fact that the actual effect of a change in any condition may be and usually is a result of numerous factors, which may entirely counteract or mask the effect of the variable being studied. The true effect of the increased temperature upon the size of the crystals may be due in large part to an actual or effective increase in the metal ion concentration, brought about by a greater degree of dissociation and by increased mobility at the higher temperature.

C. Conductivity.

In plating operations stress is frequently laid upon the conductivity of solutions and upon the use of various "conducting salts." Since the addition of almost any salts to such solutions will increase their conductivity, any such additions might be and frequently are designated as "conducting salts," although their beneficial effects may be due principally to a change in properties other than conductivity. The obvious advantage of an increased conductivity is the reduced voltage necessary to obtain a given current density. Apart from this economic advantage it is not at first evident why a well-conducting solution should produce, at a given current density, a better deposit than one with poorer conductivity. Bancroft's sixth axiom, that "treeing is facilitated by a high potential drop through the solution," suggests the advantage of increased conductivity, since at a given current density a high potential drop through the solution is due to low conductivity. If therefore two solutions could be prepared in which only the conductivity differed, this axiom could be readily tested and illustrated. Actually it is not possible, however, to change the conductivity of a solution without changing some other properties, *e. g.,* the metal ion concentration. Thus, in a copper sulphate solution the conductivity may be greatly increased by the addition of sulphuric acid, which, however, through the influence of the common ion, decreases appreciably the copper ion

concentration. Since, however, the increase in conductivity produced by the sulphuric acid is many times as great as the decrease in metal ion concentration, it may be assumed as a first approximation that the effect obtained is due principally to the increased conductivity. As it is impossible to obtain good deposits from zinc or nickel solutions containing much free acid, sulphuric acid could not be used to increase their conductivity. Sodium chloride was therefore added to these solutions for this purpose. The use of sodium chloride in such amounts was desirable merely for experimental purposes and cannot be recommended for practical work so far as any existing evidence shows. The solutions of each metal, described in Table I, were run in series until marked trees were obtained in one bath.

TABLE I.

Resistivity of Solutions.

		Resistivity at 25° C. (77° F.) Ohms
1 (a)	Copper sulphate *N* Sulphuric acid 0.1 *N*	22.4
(b)	Copper sulphate *N* Sulphuric acid 1.1 *N*	4.9
2 (a)	Zinc sulphate *N* Boric acid 0.1 *M*	32.1
(b)	Zinc sulphate *N* Boric acid 0.1 *M* Sodium chloride *N*	10.5
3 (a)	Nickel sulphate 0.8 *N* Nickel chloride 0.2 *N* Boric acid 0.1 *M*	25.8
(b)	Nickel sulphate 0.8 *N* Nickel chloride 0.2 *N* Boric acid 0.1 *M* Sodium chloride *N*	9.5

The results for copper and zinc shown in Figs. 12 and 13 illustrate very clearly the decreased tendency to treeing in solutions of high conductivity, and fully confirm Bancroft's statement. It should be noted that while the addition of sodium chloride in large amount to zinc solutions reduced the tendency to treeing, it produced a poor, spongy deposit. No definite trees of nickel were obtained in these solutions under the conditions employed, although the deposits were much smoother in the solutions containing sodium chloride.

D. Metal Ion Concentration.

Bancroft's axiom that "increasing the potential difference at the cathode decreases the size of the crystals" may be restated for practical purposes that "decreasing the metal ion concentration decreases the size of the crystals." This form of statement is based upon the usual assumption that the potential between a metal and its solution is a measure of the metal ion concentration of the solution. The two most practical methods of decreasing the metal ion concentration without changing the total metal concentration are by the addition of a common ion, and by the formation of a complex ion. Unfortunately with the first method only a relatively slight change in metal ion concentration (*e. g.*, 10^{-2} or 10^{-3}) can be produced in concentrated metal solutions, owing to the limited solubility of the salts that might be added. On the other hand, the formation of a stable complex ion usually results in a very low metal ion concentration (generally less than 10^{-8}). Consequently there is a very wide range between these two ion concentrations, which can be studied only by a careful selection of less stable complexes. If, however, the same qualitative effect is observed in each case, and if the effect is more pronounced at very low metal ion concentrations, it may be safely assumed as a first approximation that intermediate structure would be obtained with intermediate ion concentrations.

In order to obtain typical illustrations of the effect of relatively slight and very considerable reductions in metal ion concentration, deposits were produced at 1 amp./sq. dm. (9.3 amp./sq. ft.) in the following solutions:

(a) "Standard" solutions (p. 62).
(b) "Standard" solutions + *N* sodium sulphate.
(c) Normal double cyanides.

The solutions in (c) had the same metal content as in (a) and (b), and contained in each case a slight excess of sodium cyanide.

The results illustrated in Figs. 14, 15 and 16 show that in every case the addition of *N* sodium sulphate caused an appreciable decrease in the crystal size, and that with copper and zinc the formation of a complex cyanide produced a much greater change. With the nickel cyanide solution no deposit of nickel could be obtained under the conditions employed. That these effects are

due to reduction in metal ion concentration is indicated by the following approximate[6] values of the single potentials for copper measured in various copper solutions. There is no reason to doubt that similar changes in potential and metal ion concentration were produced in the other solutions.

TABLE II.

Potentials of Copper in Various Solutions.

	Potential	Copper Ion Concentration Normality
1. $CuSO_4$ — N	+0.308	0.11
2. $CuSO_4$ — N H_2SO_4 — 0.1 N	+0.304	0.09
3. $CuSO_4$ — N H_2SO_4 — 1.1 N	+0.296	0.06
4. $CuSO_4$ — N H_2SO_4 — 0.1 N Na_2SO_4 — N	+0.285	0.02
5. $CuCN.NaCN$ — 0.5 N	—0.316	$0.0_{22}1$

It will be noted that the results shown above neither prove nor contradict the possibility pointed out by R. S. Dean and M. Y. Chang,[7] that in order to produce a fine-grained deposit it is necessary to have present not only a low metal ion concentration, but also another more negative metal (considering the potential of zinc as negative) such as the alkalis or alkaline earth metals. As these were present in all the above solutions of low metal ion concentration, the results obtained may or may not have been influenced by the alkali metals.

In the above cited experiments of Dean and Chang, a comparison was made between deposits of copper produced in a solution of copper acetate-tartaric acid and in the same solution containing sodium sulphate. Similar experiments were made with solutions of normal cadmium chloride containing sodium chloride, and tenth normal cadmium chloride. There is no indication, however, that these deposits were produced at equal current densities; in fact it is stated that with copper the potential across the cell was maintained at 3 volts and after the addition of sodium sulphate the electrolysis was carried on under identical conditions. If,

[6] Measured against a normal calomel electrode but expressed in terms of the normal hydrogen electrode, assuming the value +0.286 for the normal calomel cell, and assuming that normal copper sulphate is 0.11 N in copper ion concentration.

[7] Chem. Met. Eng. (1918), **19**, 83.

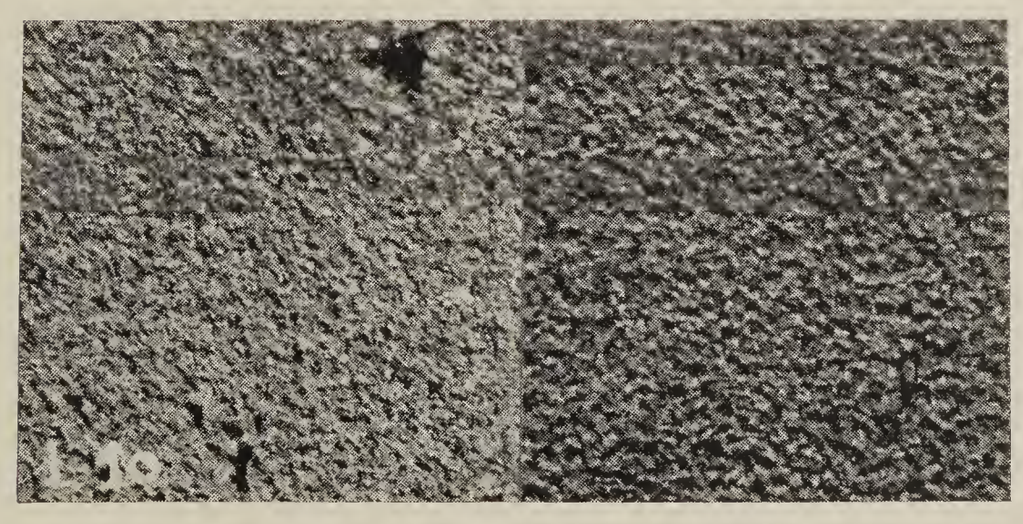
a b

FIG. 1.

Effect of Lead Concentration.

(20 diam.)

a—Deposit from normal lead solution.
b—Deposit from twice normal lead solution.



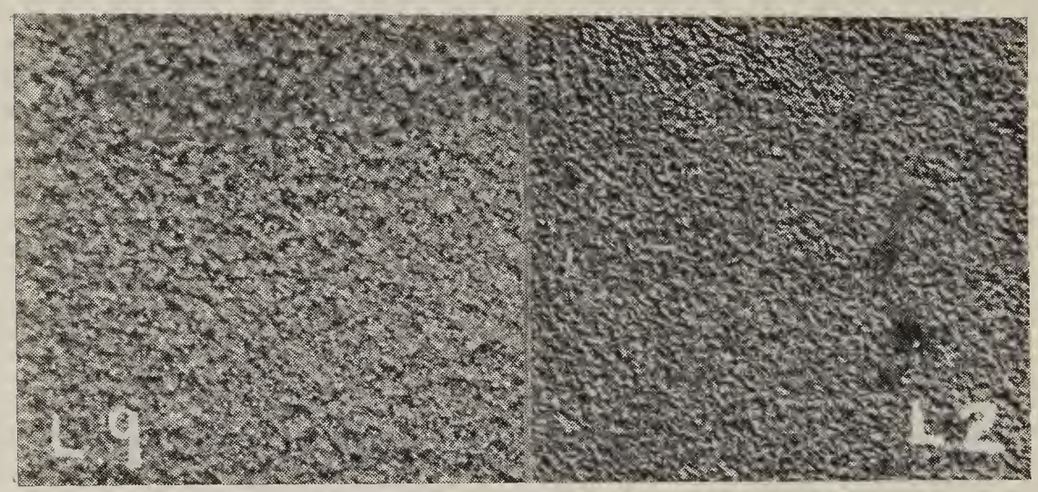
a b

FIG. 2.

Effect of Fluoboric Acid.

(20 diam.)

a—Deposit with equivalent fluoboric acid.
b—Deposit with 50 percent excess fluoboric acid.



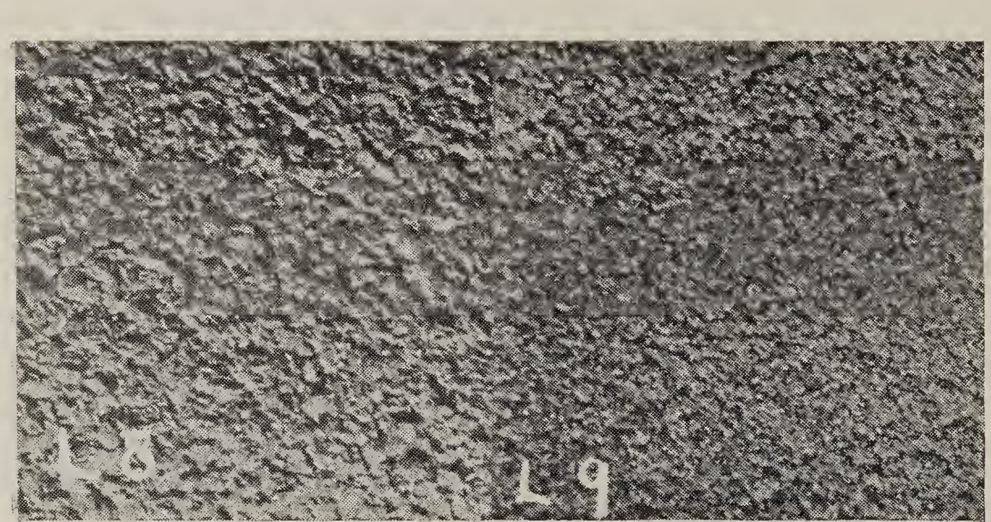
a b

FIG. 3.

Effect of Glue.

(20 diam.)

a—Deposit from solution with no glue.
b—Deposit from solution with 0.2 g/L glue.



a b

FIG. 4.

Effect of Current Density.

(20 diam.)

a—Deposit produced at 1 amp./sq. dm.
b—Deposit produced at 3 amp./sq. dm.

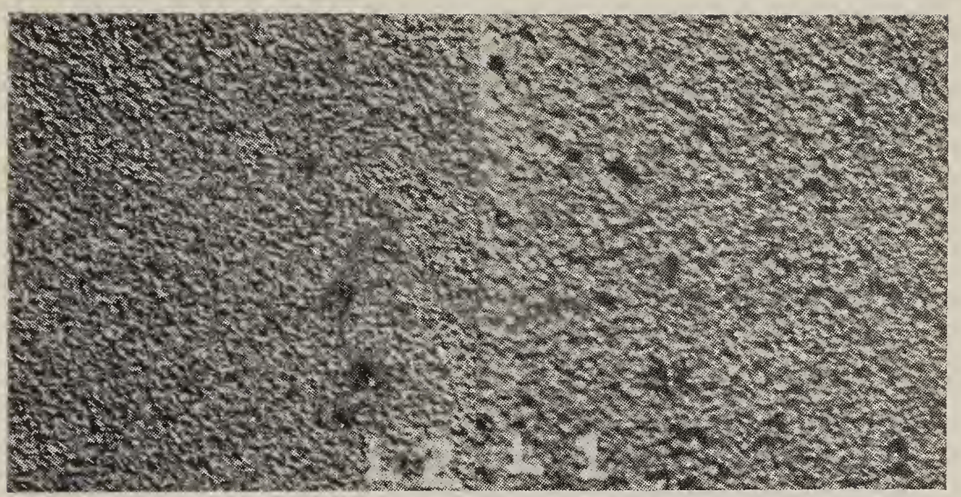


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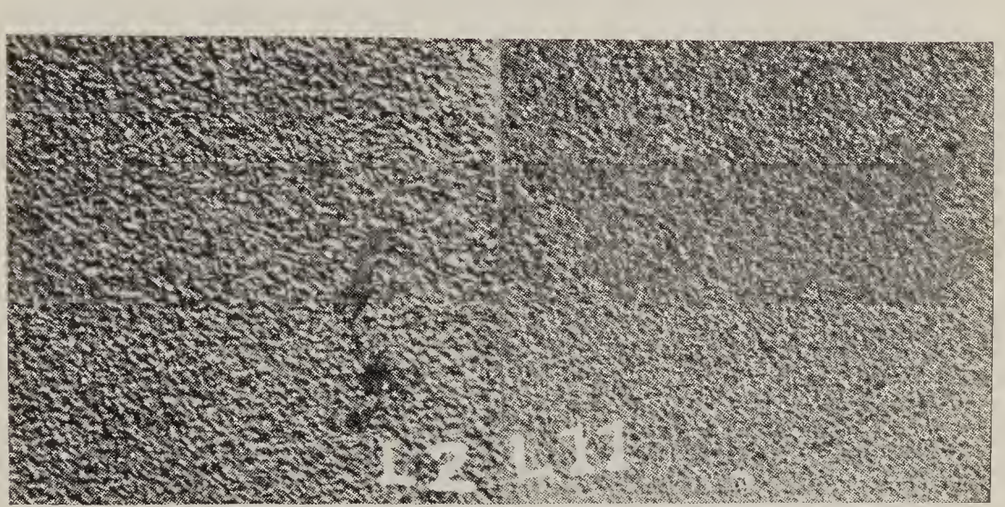
FIG. 5.

Effect of Temperature.

(20 diam.)

a—Deposit produced at 25° C.
b—Deposit produced at 50° C.



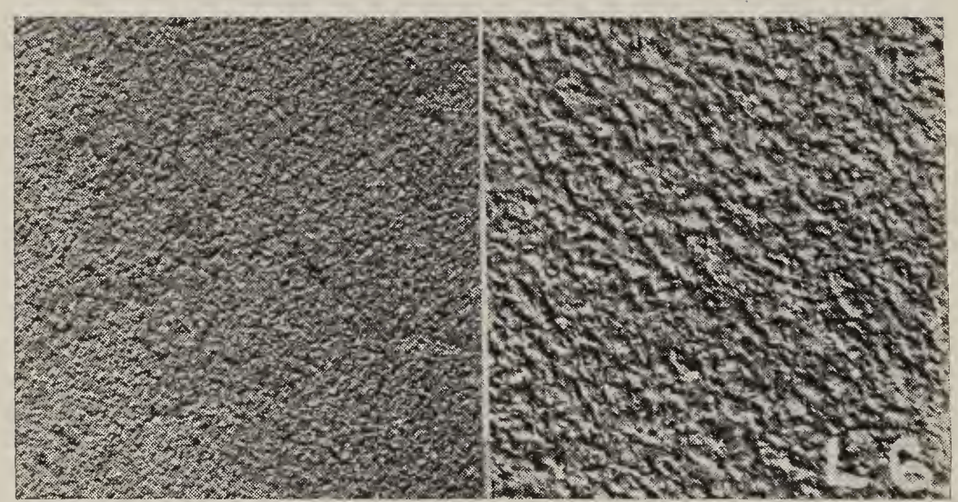
a b

FIG. 6.

Effect of Agitation.

(20 diam.)

a—Deposit from still solution.
b—Deposit from mechanically agitated solution.



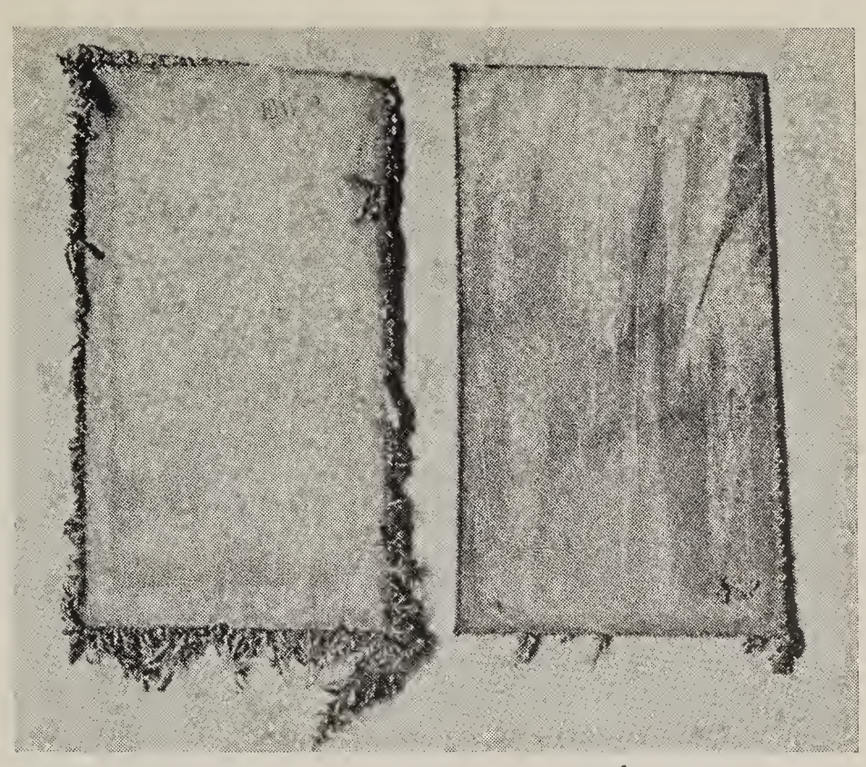
a b

FIG. 7.

Effect of Thickness on Structure.

(20 diam.)

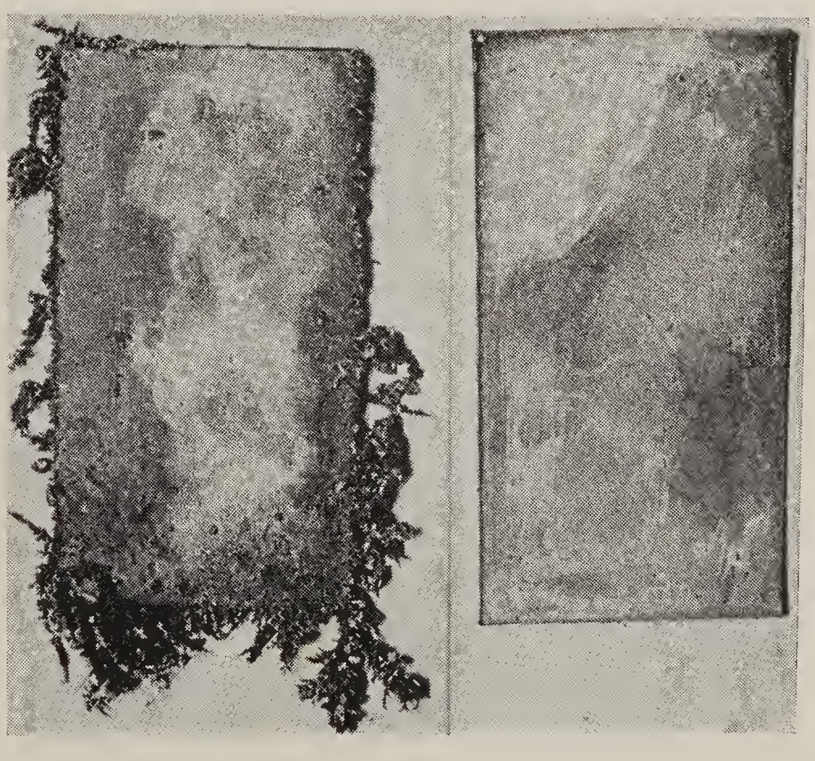
a—Deposit 0.075 mm. thick.
b—Deposit 0.25 mm. thick.



a b

FIG. 8.

Effect of Lead Concentration.
(½ size).
a—Deposit from normal lead solution.
b—Deposit from twice normal lead solution.



a b

FIG. 9.

Effect of Glue.
(½ size).
a—Deposit from solution with no glue.
b—Deposit from solution with 0.2 g/L glue.

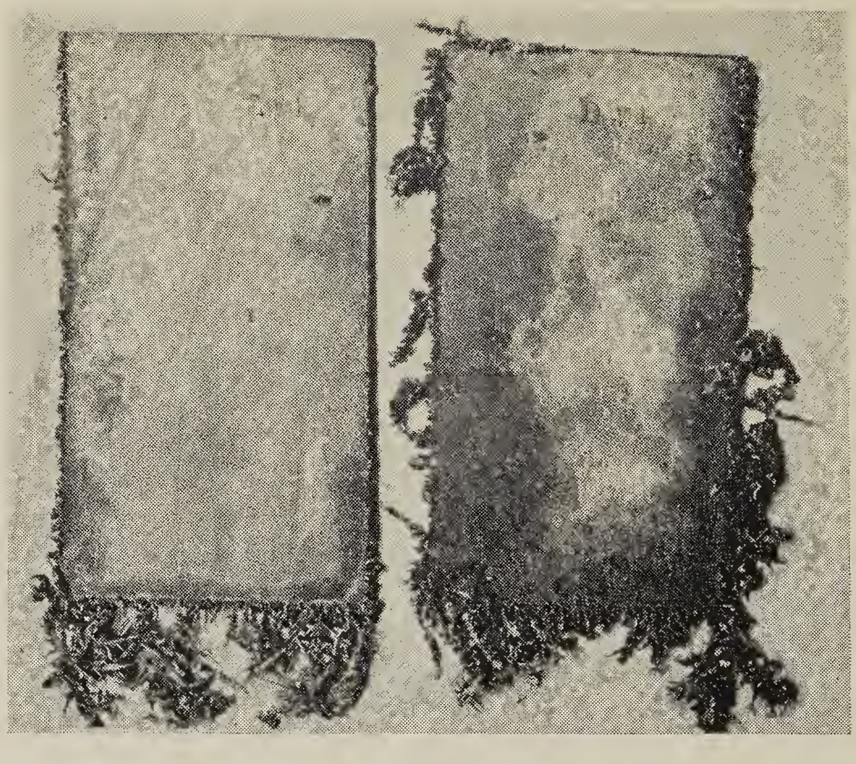


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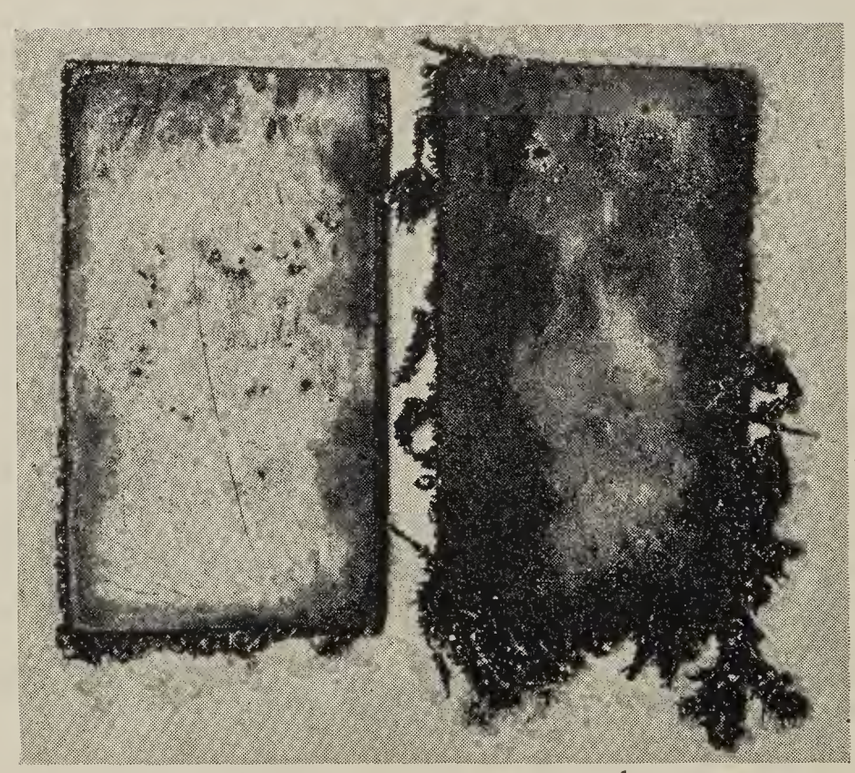
FIG. 10.

Effect of Current Density.
(½ size).
a—Deposited at 1 amp./sq. dm.
b—Deposited at 3 amp./sq. dm.



a b

FIG. 11.

Effect of Thickness on Treeing.
(½ size).
a—Deposit 0.075 mm. thick.
b—Deposit 0.25 mm. thick.

as appears probable, the voltage and not the current density was regulated, it is obvious that owing to the better conductivity of the solutions containing alkali metals, higher current densities were secured and naturally therefore finer deposits were produced.

It is evident that further work is required to determine with certainty the effect of other factors introduced incidentally with the change in metal ion concentration.

In considering and explaining to platers the apparent anomaly that for fine-grained deposits a high metal concentration, but a low metal ion concentration is desirable, it was found convenient to consider the former as a reservoir from which a small but constant supply of material available for crystal growth is furnished. Viewed from this standpoint, we can readily conceive that if the supply of metal ions is large, those crystals which through favorable size or position receive a higher current density will be able to grow more rapidly than the less favored crystals. If, on the other hand, the supply of ions is restricted, less opportunity will be afforded for excessive growth of a few crystals. That the production of a coarse-grained deposit is actually accompanied by the "crowding out" of small crystals by larger ones is evident from Fig. 4.

E. Colloids and Reducing Agents.

In spite of the many references to and applications of "addition agents" in plating solutions, no clear distinction has ever been made between colloids and reducing agents, especially in the case of such organic substances as glue or gelatin, which no doubt possess both colloidal and reducing properties. Bancroft's statement that "the crystal size is decreased when there are present at the cathode surface substances which are adsorbed by the deposited metal," evidently includes the effect not only of colloids, but also of such reducing substances as are adsorbed, or give rise to the formation of substances that are adsorbed. Wherever a substance has definite colloidal properties it appears probable that they will account for its effect in reducing the size of the crystals, especially since exactly similar though usually less marked effects can be produced by the use of inorganic addition agents such as salts of aluminum and tin, which readily hydrolyze with the formation of colloids.

The function of reducing agents in plating baths has never been clearly defined and many statements in the literature are contradictory. Assuming that the deposit will be improved in some way by the presence in the solution of a reducing agent, the effect of relatively large amounts of such substances as glucose can be accounted for. On the other hand, it is very difficult to explain, on the basis of reducing action alone, the marked and relatively persistent effect of very small additions of such substances as pyrogallol, which if they were required to cause appreciable reduction at the cathode, would be themselves rapidly oxidized. It therefore appears probable that, in many cases at least, the effect of reducing substances may be due to the formation, either directly or under the influence of the current, of colloidal particles of metal or of secondary products of decomposition of the organic substance. Thus, in the case of silver nitrate solutions, it was found[8] in a study of the silver voltameter that small amounts of both inorganic and organic reducing substances such as hydrazine, hydroxylamine, dextrose, aldehydes, phenol, hydroquinone and filter paper extract, all had the same effect, *viz.*, a diminution in the size of the crystals and the formation of striations. In all of these cases it was concluded that the effect was due to the formation of colloidal silver, the presence of which in the deposit accounted for the excessive weight of deposits from contaminated solutions.

It was not possible in connection with the preliminary experiments here described to make any actual study of the effect of various colloids or reducing substances. In order to illustrate, however, that qualitatively their effects are similar, experiments were conducted in which respectively glue (0.5 g/L) and glucose (0.2 *M* or 36 g/L) were added to the "standard" solutions. The results as illustrated in Figs. 17, 18 and 19 show that the effects are in the same direction but are usually much more marked with glue than with glucose. The deposit of nickel from the solution containing glucose was of special interest, as it was very bright and smooth. On the other hand, no satisfactory deposit of nickel could be obtained from solutions containing even as little as 0.2 g/L of glue. The deposits were bright but very brittle and possessed almost no coherence. The deposit from the zinc solu-

[8] E. B. Rosa, G. W. Vinal and A. S. McDaniel, Bureau of Standards Reprint 195.

tion containing glue was very bright but was covered with nodules and pits.

These results indicate that a great amount of research will be required before any systematic classification of the effects of colloidal and reducing substances in electrodeposition can be effected. With present knowledge it is almost impossible to predict with any probability the effect of any given addition agent in a particular solution.

5. CONCLUSIONS.

From the above-described experiments it may be safely concluded that for the common metals and over a considerable range of conditions, the axioms of Bancroft are applicable. As opportunity offers, it is hoped to conduct more exhaustive experiments in which the relative magnitude of the various effects for any given metal may be determined. From such studies it should be possible not only to define favorable operating conditions for plating baths now in commercial use, but also to suggest modifications which will permit more rapid or more satisfactory operation. By a study of the literature in the light of these axioms, accompanied by suitable confirmatory experiments, it should be possible to explain or eliminate some of the apparent contradictions in the literature of electroplating. Both chemists and platers will render great service by calling attention to apparent exceptions to these axioms, in order that if such exceptions are found to be valid, the "axioms" may be modified or added to, so as to include such cases.

6. RECOMMENDATIONS.

It should be borne in mind that even if the above axioms should be proved to be universally applicable (as is obviously not yet the case), there would still be a wide opportunity for research in the field of electrodeposition. As previously pointed out, the actual effect of any intentional or accidental change in operating conditions is almost invariably a result of a number of causes, sometimes working in opposite directions. Before it is possible, therefore, to define the best working conditions for any specific plating operation or to predict the effect of a given change, it is necessary to determine not alone the direction (which for most

cases may be indicated by Bancroft's axioms) but also the magnitude of each effect for a particular metal or solution. In addition it is necessary to study those specific properties of the metals or solutions which may exert effects independent of the above or any similar generalizations. Finally, even after favorable operating conditions are defined, it is essential to develop simple methods of testing solutions and deposits, in order that such conditions may be maintained.

In view of the large field still to be studied, it would be premature to formulate any detailed general operating conditions with the idea that they would be actually applicable in every possible case. Nevertheless it appears desirable at this time to restate, in the form of suggestions rather than advice, to platers and others interested in plating, the general principles which will probably govern the choice of operating conditions. It is recognized that these suggestions by no means cover all the problems of electroplating, and that they should be used only as a guide, and with care and discretion. Probably their chief value at this time will be not so much in developing any radical modifications or new solutions, as in indicating the direction in which any desired improvement in existing operations may be sought. Where examples are given, they are used solely as illustrations of the particular factor and are not intended as directions for that particular solution. Where information is lacking the need is indicated.

A. Composition of Solutions.

(1) *Choice of the metal salt.* For purposes of discussion we may consider the metal salt as the compound present in the solution which contains the major part of the metal to be deposited, *e. g.,* copper sulphate, zinc chloride, sodium silver cyanide, etc. It is therefore immaterial (except from the standpoint of convenience and economy) whether the salt is purchased and added as such or whether it is formed in the solution. Thus a solution of sodium silver cyanide may be prepared from either silver cyanide or silver chloride, the only difference being that in the latter case the resultant solution contains in addition an equivalent amount of sodium chloride. In some cases the electrolyte may be composed of nearly equivalent quantities of two metal compounds,

as when zinc oxide is dissolved in sodium cyanide, forming sodium zinc cyanide and sodium zincate.

The compound selected (or its solution) should have as many as possible of the following properties. It should be borne in mind that the actual choice is almost invariably a compromise. Where some of the desirable properties are lacking they may be supplied by appropriate additions to be considered later.

(a) High solubility in order to yield a high metal concentration.

(b) Good conductivity.

(c) Stability, *i. e.*, resistance to hydrolysis (formation of basic salts), and to oxidation or reduction.

(d) A low metal ion concentration, such as is possessed by complex cyanides.

(e) The ability to corrode the anode nearly quantitatively. This is required especially for metals such as nickel which have a tendency to anode passivity.

(f) Good throwing and covering power. The conditions which determine the "throwing power" of a solution have never been clearly defined and are in need of study. They are no doubt closely connected with the conditions which determine the crystalline structure.

(g) A sufficiently low price to permit economic operation in the light of its relative advantages.

(2) *Additions to solutions.* It has been frequently maintained that the simplest solution is the best plating solution. Consideration of the principles discussed in this paper shows clearly that this is by no means always or usually the case, and that certain additions to solutions produce marked improvement. On general principles, however, and in view of the desirability of using solutions that can be readily analyzed and controlled, it cannot be too strongly emphasized that no additions to plating solutions should be made unless their presence has been shown by experiment to be distinctly beneficial in one or more respects. In this connection it is important to note that by the methods of preparation of solutions frequently employed, substances other than the major metal compound are often produced in the solution; thus sodium chloride by the solution of silver chloride in sodium cyanide, sodium carbonate by the solution of copper carbonate

in cyanide, and sodium hydroxide by the solution of zinc oxide in cyanide. Whether in any particular case the presence in such amounts of the compounds thus formed is an advantage or not, can be determined only by investigation. It is manifest, however, that if their presence is desired, it is immaterial whether they are added to or produced in the solutions. Thus, there is no advantage, other than possible convenience or greater purity, in substituting a metal cyanide for a carbonate and then adding an alkali carbonate to the solution. Where, as in the case of copper, a change in valence is required to produce a cyanide solution, the use of a metal cyanide, or else of a reducing substance cheaper than sodium cyanide, is desirable.

It should be noted that frequently a given addition may accomplish several purposes and in other cases it may improve one property at the expense of another. Wherever it is possible to accomplish several purposes with one substance, it should be used rather than several different substances. Among the purposes for which substances may properly be added to a solution are the following:

(a) To increase the solubility either of the metal salt or of the oxides or basic compounds that may be formed. Thus the addition of sodium chloride increases the solubility of nickel sulphate, and the presence of sulphuric acid in a copper sulphate solution prevents the formation of basic cupric or insoluble cuprous compounds. Similarly the presence of excess cyanide prevents the precipitation of insoluble cyanides.

(b) To increase the conductivity. This may be accomplished by the addition of an acid to acid solutions such as copper sulphate, of a base to alkaline or cyanide solutions, and of a well-conducting salt to solutions which must be kept nearly neutral.

(c) To regulate the acidity or more specificially the hydrogen ion concentration of such solutions as zinc and nickel, which must not have excessive acidity. For this purpose weak acids (or their salts) may be used, such as acetic and boric acids.

(d) To reduce the metal ion concentration. In the case of simple solutions this may be effected by the addition of a salt with a common ion, *e. g.,* sodium or potassium sulphate to metal sulphate solutions. If we consider a complex salt, such as a

double cyanide, as the major compound, it already has a low metal ion concentration which can be further reduced by the addition of an excess of cyanide.

(e) To aid anode corrosion and decrease the polarization resistance, for example, the addition of nickel chloride or alkali chlorides to a nickel sulphate solution and the addition of hydroxides or cyanides to an alkaline zinc solution.

(f) To increase the throwing power. As previously stated, this property has never been thoroughly studied.

(g) To form colloids (or other dispersoids) which will migrate to the cathode and reduce the size of the crystals. These may be added directly in the form of glue, gelatin, casein, albumen, etc., or in the form of substances which will yield colloids by hydrolysis or reducing action.

(h) To exert a reducing action at the cathode or in the solution. This may possibly be associated with (g).

(i) To yield ions of a more negative metal (such as sodium, potassium, magnesium, etc.). Whether this effect is connected with (d) has not been definitely determined.

B. Current Density.

The highest current density should be employed that will yield satisfactory deposits (unless it is intentionally desired to obtain coarse-grained or soft deposits). The permissible current density may be increased by suitable agitation of the solution, especially the portion of the solution close to the cathodes.

C. Temperature.

If fine grained, bright, or hard deposits are desired, the temperature should be kept as low as convenient, if necessary by using artificial cooling. If coarse or soft deposits are desired the temperature may be allowed to rise. Since the effects of increased current density and temperature are in opposite directions, it is possible to obtain in warm solutions at a high current density (which at a given voltage may be more readily secured in warm solutions) as fine deposits as are produced in cold solutions with a lower current density.

7. SUMMARY.

Experiments have shown that Bancroft's "axioms of electroplating" are applicable over a wide range of conditions and with a variety of metals. They may, therefore, serve as a valuable guide in plating research and in practical plating operations.

In conclusion the author desires to acknowledge his great obligation and appreciation for the services of his associates who prepared the numerous solutions, deposits and photographs involved in this study, especially Zalia Jencks, L. B. Ham, S. C. Langdon, H. E. Haring and L. D. Hammond.

U. S. Bureau of Standards,
Washington, D. C.

DISCUSSION.

G. B. HOGABOOM[1]: This paper is of great interest to electroplaters, and Dr. Blum should be commended for proving Dr. Bancroft's axioms. The plater has never explained the real effect of different current densities, or the changes due to temperature. The microphotographs so well illustrate the work done, that even those without technical knowledge can readily understand the text. We need more papers like this one, papers that have a practical and commercial application.

F. C. MATHERS[2]: I wish to discuss the effects of addition agents and the important part played by the acid ion or radical in combination with the metal which is being plated. The details that Dr. Blum has worked out are very necessary and very useful, but there are a number of other things that have important actions in plating solutions. Scientists often scoff at some of the weird things that are recommended for use in plating solutions. However, I feel that the unusual substances may have wonderful effects and I do not care to condemn any of them. I have brought some samples of cathodes that show the important relations between the addition agent and the acid ion in the bath. A stannous sulphate bath gives a smooth, firm, finely crystalline deposit,

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[2] Asst. Professor of Chemistry, University of Indiana.

while a stannous chloride bath gives a loose, crystalline deposit, although both have the same concentration, same acidity, same addition agent, and are operated under the same conditions. I have been unable to obtain anything except a loosely crystalline deposit from the stannous chloride bath. Many chemists think that chlorides are bad in plating solutions. These samples illustrate one case where the chloride bath is worthless unless a new addition agent is discovered.

Glue is commonly considered to be the proper addition agent for plating baths. It does work very well in the case of lead, but with many metals other addition agents are superior. Valuable addition agents are not confined to the class of substances that we ordinarily call colloids. Silver nitrate solution containing pyrogallol, which may function as a colloid, gives a very fluffy loose cathode deposit. I have a tube filled with these crystals, which were easily shaken from a cathode. Tartaric acid added to the silver nitrate solution causes the cathode to be very hard and firm with no indication of a rough crystalline structure. Other substances have a similar action. If ferric nitrate is added along with the tartaric acid, the deposit is black, shiny, and contains iron. Glue has some action in the silver nitrate bath, but it is very slight compared with that of the tartaric acid. Metaphosphoric acid is about as good as the tartaric acid. The colloidal nature of these substances is doubtful. There is no doubt but that the tartaric acid is taken up by the deposit. Here is a cathode that was cut in two and one piece ignited. The heated portion has swelled or enlarged. The odor of burning tartaric acid was noticeable during the heating.

Here is an antimony deposit from a fluoride bath without an addition agent: it is firm but crystalline and rough. This deposit is from the same bath except that the addition agent, aloin, has been added: it is very smooth and free from crystals. The further addition of a little clove oil gives the cathode a polished, glossy appearance. It is impossible to get cathodes like these out of chloride solutions.

W. D. BANCROFT[3]: I think there has been a very natural but unfortunate confusion in the use of terms. Mr. Mathers was speaking about whether a thing was a colloid or not. It is not

[3] Professor of Physical Chemistry, Cornell University.

of the slightest importance whether your addition agent is a colloidal solution or a true solution. The important thing is to have your metal come down more finely divided, and it is essential that your addition agent should be precipitated with the metal and should cut down the size of the crystals. That happened originally and is more apt to happen in the case of a colloidal addition agent, but it is not at all necessary; just the same as we can get a colloidal precipitate from a sugar solution, and nobody claims that the colloid is in the sugar solution. We started with the most obvious case and then used our terms unfortunately, so that now we have got around to the belief that the addition of colloids is the essential thing and the question whether it dissolves or not is important, whereas it is of no importance at all.

The difference between stannous chloride and stannous sulphate was very interesting indeed. I should like to ask Mr. Mathers how he accounts for the difference.

F. C. MATHERS: I think that probably there is some kind of a complex combination in the bath, and we do not get that complex in the case of the chloride.

W. D. BANCROFT: A lower concentration of tin?

F. C. MATHERS: I do not know. The only thing on which I am basing my theory is the fact that some of these things that affect the deposit, the phosphoric acid and tartaric acid, are quite likely to give complexes.

W. D. BANCROFT: There you have a nice, clear-cut case, stannous chloride and sulphate, and that is why we should find out whether it is due to adsorption by the cathodes of sulphate or phosphate in some form or other, or what is the cause.

WILLIAM BLUM: Just as a suggestion, without any attempt at a definite explanation: if, as is probably the case, the sulphate is more hydrolyzed than the chloride, we would have a greater opportunity for the presence of colloidal stannous hydroxide in the sulphate solution. The fact that inorganic colloids do affect the structure of the deposit is well borne out by the behavior of stannic and aluminum sulphates in the acid copper solution, both of which act as inorganic colloids. In this case I would suggest offhand that the stannous hydroxide, if present, may be acting

as a colloid in connection with the deposition of tin, and there may be a better opportunity for it to form in the sulphate solution than in the chloride. If such were the case, the inorganic colloid might augment any beneficial effect of the addition agent which Dr. Mathers added; and thereby account in part for the better deposit in the sulphate solution. In any case, the above example is by no means a simple one, and illustrates the present uncertainty as to the effect of any particular addition agent rather than any intrinsic effect caused by the difference in the acid radical in simple solutions free from addition agents.

F. C. MATHERS: If one adds sodium chloride (NaCl) to the stannous sulphate bath, it spoils the deposit. That would not be in line with your theory.

COLIN G. FINK[4]: I would like to ask Mr. Mathers whether he did not find that in the stannous chloride baths, the addition of sodium chloride improved the deposit, although it spoiled it in the case of the sulphate? What other cathode besides tin is present in the sulphate solution?

F. C. MATHERS: I had no other; these were pure solutions of stannous chloride.

COLIN G. FINK: Why not substitute tin for platinum in dilute sulphuric acid?

F. C. MATHERS: I never tried it in very dilute acid; I used probably two percent of free acid and four percent of tin.

ANSON G. BETTS[5] (*Communicated*): Perhaps the best way to bring out a consideration of importance in this subject is to refer first to the familiar explanation of the formation of rain from clouds and mist, according to which the growth of some of the fine drops at the expense of the still smaller ones continues until the drops are large enough to fall as rain. The explanation is that the surface tension of the smaller droplets is less than that of the larger ones, by reason of greater curvature, and in consequence the pressure of the aqueous vapor is greater from the small and more highly curved surfaces than from the others. The medium is supersaturated with aqueous vapor as compared

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[5] Nyack, N. Y.

with big drops and unsaturated as compared with the little ones, so that evaporation takes place from the one and condensation from the other until the process is interrupted by the falling of the rain. The result is a reduction of the total area of surface of a given amount of water, the advancement of the system to a more stable condition.

Another familiar effect of surface tension, applying to a solid in a liquid medium, and consequently coming nearer to our subject, is what takes place when we heat a precipitate of barium sulphate to make it coarser and better for filtering. The solution is supersaturated at the comparatively flatter surfaces of the larger particles and unsaturated at the others, which causes the growth of the larger particles and the disappearance of the others. The change is quite rapid because the particles are very small and the effect of surface tension is proportionately great.

A consideration of this relative instability of sharply curved surfaces or sharply bent surfaces as compared to plane surfaces, and further, concave surfaces, is valuable in connection with the author's subject, and is the basis of a workable theory which fits the facts.

Given a metal surface under the conditions of metal plating, the effect of the surface tension of the metal is in the direction of producing and maintaining a smooth surface. The effect of the surface tension is to oppose the deposition of metal on projecting points and crystal angles and to assist in filling the hollows in an entirely analogous way. Without it there could be nothing more than a crystallization into a non-solid mass, aggravated by the more rapid deposition of metal on the exposed points at least distance from the anode, and the rapid growth of "trees." The case is not exactly parallel to the case of the water mist and the barium sulphate, as in the latter two, fixed quantities of material were considered, and in this case we have an increasing amount of material, but nevertheless, the surface tension is active in producing a product with the minimum amount of surface, the most stable condition, the condition of least energy, that is, the smooth surface. Of course, surface tension is potent on a very small scale, but rough metallic deposits, and non-solid deposits begin on a very small scale, the roughnesses becoming rapidly aggravated to a visible scale.

If this is so, we might expect metals with high surface tension to yield solid deposits. We can expect the surface tension to be greatest in the strong, hard metals, and find that these are the metals yielding the best deposits, whereas the weak metals, lead and tin, are prone to "treeing." Further, we would expect, if we could make a lead deposit hard, it ought to be smooth. This is what we do when we add glue to the lead solution. The deposit is much harder and stronger than ordinary lead.

Further, if the above is correct, we should also examine the anode surface, also in a condition of flux, and see if the surface tension is not making the projections dissolve faster than the hollows in accordance with the theory. I have found this to be the case. Two lead solutions were run side by side, one containing glue and yielding a solid deposit, and the other without glue yielding a non-solid deposit, and it was found that the anode surface in the former was visibly smoother.

The effect of immersion of a metal in a plating bath should be to reduce the surface tension, and consequently whatever differences exist in the surface tension of two metals, would be greater in degree when immersed in their solutions. These surface tensions should be measurable by methods analogous to those used for liquids. I have made attempts to get that of copper by reading the E. M. F. between a fine copper wire and a heavy wire immersed in copper sulphate, but my apparatus was not sufficiently delicate, and it would be very important to have both pieces of the same purity and the same molecular condition, that is, of the same degree of hardness.

The author discusses the six "axioms" of Prof. Bancroft, but does not seem to be able to prove very much for them. The first two merely state that it is necessary to have the right kind of a solution.

In dictum No. 3 there is a chance for confusion, because there seems to be an assumption that the surface of an electro-deposit is a crystalline surface, when, although it may give a vague impression of a mass of individual crystals, the assumed crystals are nothing but a series of bumps and nodules. A coarsely crystalline deposit with a smooth botryoidal exterior is a better deposit for practical purposes than a fine felting of individual crystals with interstices containing electrolyte. The surface shown by

the author, Fig. 2*a*, certainly does not look to be more crystalline than *b*, and *c*, but merely has a coarser surface. The lumps shown do not look like individual crystals. His deposits shown in Fig. 3 would not appear to bear out Prof. Bancroft's contention. This third dictum does not appear to be proved, and if it were, it should be made clear as to whether it refers to the crystalline grain of the deposit, or to the coarseness of the surface. Furthermore, electro-plating is never a 100 percent process in its simplicity. Other phenomena take place; the dissolving effect of the oxygen of the air, evolution of hydrogen and other gases consuming current, passage of small currents without electrolysis, secondary electrolysis, etc. If we work with very small current densities, we can get irregular results, or none, from the comparative magnification of these factors, so results from very low current densities may be entirely misleading and not safe to base this theory on. Within the working ranges, I believe it is generally found that excessive current densities give inferior or bad results, and consequently I cannot accept the above dictum as likely to be found correct. Also, the beneficial effect of the surface tension has relatively less play at higher current densities, and therefore higher current densities would favor rough deposits and "trees," as is a fact. Increasing the conductivity of the solution assists the beneficial effect of the surface tension, as it lowers the opposing force, namely, difference in voltage drop to the ridges and depressions of the deposit. For the above reasons dictum No. 3, while it may or may not be true as to crystallization, is misleading unless a distinction is clearly made between interior crystallization and surface bumps.

Coming to the fourth "axiom," the same distinction is important, but the "adsorption" referred to is something not proved as far as I am aware. The use of glue in a lead bath makes the deposit smooth, but on account of the effect on the anode surface the vague suggestion of "adsorption" would be hard to develop into a clear explanation of both anode and cathode observation.

The fifth and sixth rules are at least in some respects contradictory. No. 5 cannot be strictly true as stated, as the current density is itself one of the conditions and cannot be increased and kept the same at the same time, and certain other factors, as voltage drop, are dependent on, and variable with, the current

density. If the current is increased, the voltage drop either has to increase, or temperature change to increase conductivity, or strength of solution change, and it is not clear as to what compensations could and could not be made under the definition of "conditions." No. 6 contradicts No. 5, in that No. 5 states that no harm is done by increasing the current density, which practically means an increase in potential drop, and No. 6 states that this potential drop does do harm, in increasing the tendency of "treeing."

The surface tension theory presents something tangible, in that surface tension is a force that we know exists, and while perhaps it might not be expected that a piece of rough metal immersed in a suitable electrolyte would in a measurable time smooth its surface out, it should be remembered that an electro-deposit is a series of surfaces one built on another, and each one subject during the period of its formation to the well-known laws of surfaces, and that the surface tension, in proportion to the amount of material involved, is a very considerable force.

A good deal has been said about colloids, dispersoids, etc., but the trouble with this convenient theory is that very effective results can be secured with substances that do not appear to have any colloidal properties at all.

A phenomenon of significance in electro-plating is that there is an apparent disappearance of energy. Two copper plates are placed in an acid copper sulphate solution. The conductivity of the solution is carefully measured, and a certain amount of current established, and according to Ohm's law a certain voltage results, but in fact the voltage is found to be greater, so that energy is being absorbed in a manner not explained by Ohm's law, and the electric current is doing other work of some kind, beside overcoming the resistance of the solution and electrodes. Air being excluded, and the copper being pure, the solution remains unchanged, so that work of some kind is being expended on the copper, which may or may not be finally discharged as heat —it would take careful calorimetric experiments to determine. In the case of antimony deposited from the chloride solution, the deposit is said to be explosive, showing absorption of energy. Less amounts of extra energy may be stored in ordinary deposits.

As the author suggests in his introduction, investigations along

the lines of general principles are needed, and it is submitted that such studies should not overlook the consideration of the forces and energy requirements involved.

WILLIAM BLUM (*Communicated*): Dr. Betts has, in his discussion, very properly called attention to the limitations of Bancroft's axioms, or other generalizations upon electro-deposition. No doubt a more fundamental basis for such generalizations must include a thorough consideration of the energy changes involved, some of which are of considerable magnitude. As substantiating the expenditure of energy during deposition in forms other than heat, I may cite the fact that it has been found possible to produce very decided bending of a case-hardened steel plate one-quarter inch thick, by depositing nickel or copper upon it.

As a basis for any such fundamental study it is necessary to acquire more actual facts about electro-deposition. Intelligent application of Bancroft's axioms and similar principles will lead to the acquisition of such definite information as is now lacking, by permitting the prediction of the effect of many changes, at least over a wide range.

On pages 215 and 216 of the paper it was pointed out by the author that these axioms are by no means exhaustive, and that (with the possible exception of No. 6) they refer primarily to the size and shape of the actual crystals and not to the causes of such defects as pits, ridges, cracking, curling, etc. Thus on page 219, it was pointed out that the roughness in Fig. 2*a* to which Dr. Betts refers is probably due to the effect of hydrogen bubbles. Entirely apart from this effect, however, the individual crystals in 2*a* are larger than those in 2*b* or 2*c*. Similarly in Fig. 3, the crystal size diminishes from 3*a* to 3*c*, which latter represents a mirror-like deposit (except for the polishing scratches). 3*d* is an example of a badly curled cracked nickel deposit, in which, however, the crystals are so fine as to be invisible with the magnification used. It should be noted that these experiments were conducted not, as Dr. Betts apparently assumes, with low current densities, but with current densities approaching or exceeding those actually usable in commercial practice. The "working range" is the range within which good commercial deposits can be secured, and there is absolutely no question that w i t h i n

this range, increasing the current density yields finer grained deposits (although not necessarily smoother).

Dr. Betts's statement that "the adsorption referred to is something not proved," overlooks the very large amount of experimental evidence that from solutions containing organic materials which act as "addition agents" the deposited metal contains appreciable amounts of organic matter. Whether its presence there is caused by "adsorption" is largely a question of definition.

Axiom No. 5 would be more explicit if for "condition" the term "composition of solution" were substituted, since it is primarily the depletion of the solution adjacent to the cathode which limits the applicable current density. Similarly axiom No. 6 is of interest principally in connection with the effect of the potential drop required to produce a given current density, in other words in connection with the resistivity of the solutions. With these limitations, the present apparent contradictions between axioms 5 and 6, to which Dr. Betts refers, disappear.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

LEAD PLATING FROM FLUOBORATE SOLUTIONS.[1]

By W. Blum, F. J. Liscomb, Zalia Jencks and W. E. Bailey.[2]

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1. INTRODUCTION.

In June, 1918, a demand for lead plating arose in connection with the lining of gas shells and the coating of the boosters and adapters used in gas shells. Upon inquiry, it was found that lead plating was not in extensive commercial use, almost the only application at that time being the coating of steel storage battery fittings in a few plants. In most of these plants the fluosilicate solution as used in lead refining was employed, although the fluoborate solution was in successful use in at least one plant. The perchlorate bath, the only other solution recommended for plating, had not found commercial application.

[1] Manuscript received July 31, 1919.

[2] Published by permission of the Director of the Bureau of Standards.

In order to meet the immediate military needs, a preliminary circular on lead plating was prepared by the Bureau of Standards, in which simple directions for lead plating were given. Comparison of the fluosilicate and fluoborate baths showed that the fluoborate possessed certain advantages, among which were:

(1) It may be prepared by any operator from chemicals easily obtainable; (2) it may be used to plate directly upon steel, whereas in the fluosilicate solution the steel must be first copper plated or receive other special treatment; (3) the fluoborate solution is less readily decomposed and produces less sludge than does the fluosilicate, and (4) the deposits from the fluoborate solutions are more dense and more nearly impervious than those obtained under similar conditions from the fluosilicate solutions.

While there was no occasion to question the usefulness of the fluosilicate solutions then employed (which can on a large scale be prepared more cheaply than the fluoborate), the use of the fluoborate solutions was recommended in the preliminary circular.

Since that time, a very considerable amount of lead plating was conducted[3] from both fluosilicate and fluoborate solutions upon gas shells and boosters. Another extensive application of lead plating was for the purpose of bringing up to weight, underweight shells. This was readily accomplished by depositing upon the inside of the shell, the requisite weight of lead, which with larger shells amounted in some cases to several pounds. By this means it was possible to salvage large numbers of shells which would otherwise have been rejected.

In view of the marked advantages of the fluoborate solutions, special attention was given to them in the investigations conducted by the Bureau. In order to obtain the most information of immediate value and to define the most favorable conditions, the early experiments were confined to a study of the effects of the various conditions of operation. It was not possible at that time to conduct any extended investigation of the fundamental principles, or of the possible addition agents other than glue, which might be used in such solutions. Subsequently a few more carefully controlled experiments were conducted from which more nearly

[3] A. G. Reeve: Trans. Amer. Electrochem. Soc. (1919), **35**, 389.

quantitative conclusions might be drawn. The work as described in this paper represents therefore the result of a large amount of preliminary work and of a comparatively few well-defined experiments upon the effects of changes in the composition, concentration and current density, together with some data upon methods of testing and analyzing such solutions. No attempt was made to secure high accuracy in the various measurements, since only the more pronounced effects of the different variables were being investigated. While it is not possible to predict at this time the future scope or applications of lead plating, it seems desirable to place on record the results of these experiments, which it is not practicable to continue at this time.

2. HISTORICAL.

Although a large number of solutions have been suggested and tried for the deposition of metallic lead, the only ones which have proved to be commercially successful are the fluosilicate and fluoborate. As F. C. Mathers[4] has published an extensive bibliography upon the deposition of lead from its various solutions, no reference need be made in this paper to the literature, except so far as it relates to the solutions of the fluorine acids.

The first electrodeposition of lead from fluoborate or fluosilicate solutions was conducted by Leuchs,[5] who suggested such solutions for the production of galvanoplastic deposits, or the preparation of pure metals, including copper, zinc, lead and silver. Betts secured a patent[6] for the process of refining lead by electrolysis with substantially the same electrolyte as was proposed by Leuchs. Subsequently[7] Betts patented the electro-refining of lead, using lead salts of fluorine acids, with the addition of an "agent capable of restraining the crystallization of the deposit." For this purpose he suggested the use of gelatin, pyrogallol, resorcinol, saligenin, orthoamidophenol, hydroquinone, and sulphurous acid. It is interesting to note that although he considered the improvement "to be due to the reducing action of the

[4] Trans. of the Am. Electrochem. Soc. (1913), 23, 153-192.

[5] German patent 38,193; 1886.

[6] U. S. Patent 679,824; Aug. 6, 1901.

[7] U. S. Patents 713,277 and 713,278, originally issued Nov. 11, 1902, were re-issued respectively as Re-issue 12,301 on Jan. 3, 1905, and as Re-issue 12,117 on June 9, 1903

agent employed," he obtained best results with gelatin; a substance characterized more by its colloidal properties than by its reducing action, especially when present in so small a concentration as that recommended by Betts, one part by weight in five thousand parts of solution. Substances such as glue or gelatin have been frequently employed in plating solutions to reduce the size of the crystals.[8] Fluosilicate solutions containing glue or other addition agents have been extensively used for lead refining, but only to a slight extent for lead plating previous to 1918, although Betts stated that his method was "well adapted for electroplating metal bodies with lead."

It is of interest to note that Betts and Kern[9] recommended the use of lead fluosilicate solution as a coulometer bath; and Fischer, Thiele and Maxted[10] considered the fluoborate solution most suitable for a lead coulometer. These conclusions regarding fluoborates are verified by the experiments conducted here, in which, as compared with a copper coulometer, cathode efficiencies of from 99 to 100 percent were almost invariably obtained.

3. GENERAL PRINCIPLES OF FLUOBORATE SOLUTIONS.

When hydrofluoric acid and boric acid are mixed, it is generally assumed that fluoboric acid, with the formula HBF_4 is formed, according to the simple equation:

$$4HF + H_3BO_3 = HBF_4 + 3H_2O.$$

There is abundant evidence[11] to show that the reaction is by no means as simple as this, and that the various other compounds which may be formed, depend upon the concentration, temperature and length of time after the mixing. It has been definitely established, however, that in concentrated solutions (such as are employed in plating) at least four moles of hydrofluoric acid combine with one of boric acid, to form an acid stronger (chemically) than the original hydrofluoric acid, as indicated by its

[8] H. Jacobi and Eugene Klein: U. S. Patent 82,525, Sept. 29, 1868, added gelatin to iron solutions to improve the texture of the iron deposit.

[9] Trans. Amer. Electrochem. Soc. (1904), 6, 67.

[10] Z. Anorg. Chem. (1910), 67, 302 and 339.

[11] Abegg: "Handbuch der Anorganischen Chemie," Vol. III—1, pp. 20-23. Gmelin-Kraut: "Handbuch der Anorganischen Chemie," Vol. I—3, pp. 452-5.

greater conductivity and its increased hydrogen ion concentration. Experiments conducted by Mr. A. D. Bell at this Bureau with a hydrogen electrode have shown that on the addition of boric acid to hydrofluoric acid, there is an increase in hydrogen ion concentration, with a maximum at a point corresponding to about five moles of hydrofluoric acid for one of boric acid. However, in order to avoid any uncertainty in the preparation of the solutions, the simple formula HBF_4 has been adopted. Thus, if a solution is stated to be $3N$ in total fluoboric acid, it has been prepared by mixing twelve moles of hydrofluoric acid and three of boric acid, and diluting to one liter, upon the assumption that three moles of a monobasic acid (HBF_4) have been formed.

In considering the neutralization of fluoboric acid to form salts, there is even greater uncertainty owing to the fact that some of the complex acids which are probably formed by hydrolysis may be dibasic. Actually it has been found that when a normal solution (as above defined) of fluoboric acid is saturated with basic lead carbonate, the lead content of the resulting solution is about $1.2N$, indicating that more lead is dissolved than corresponds to the simple formula $Pb(BF_4)_2$. It has therefore been found most practical to define the composition of the solutions in terms of normal concentrations of (a) lead, (b) total fluoboric acid (HBF_4), and (c) excess boric acid considered as a monobasic acid (over and above that required to combine with all the hydrofluoric acid to form HBF_4).[12] This may be conveniently illustrated by a solution defined as

Lead — normal
Fluoboric acid — normal
Boric acid — 0.2 normal

Such a solution would contain 103.6 g/L (13.9 oz./gal.) of lead, which corresponds to 129 g/L (17.3 oz./gal.) of basic lead carbonate, assuming the formula $2PbCO_3.Pb(OH)_2$. It would also contain 88 g/L (11.9 oz./gal.) of fluoboric acid HBF_4 the formation of which would require 80 g/L (10.7 oz./gal.) of 100 percent hydrofluoric acid HF (or a corresponding amount of acid

[12] Since lead fluoride is precipitated from solutions containing any appreciable excess of hydrofluoric acid above that required to form HBF_4, no such lead solutions can be prepared.

of any given concentration, *e. g.*, twice the above amount of 50 percent hydrofluoric acid), and 62 g/L (8.3 oz./gal.) of boric acid. In addition it would contain 12.4 g/L (1.7 oz./gal.) of excess boric acid. The actual preparation of such a solution would therefore require:

	g/L	oz./gal.
Basic lead carbonate.....	129	17.3
50 percent Hydrofluoric acid.....	160	21.5
Boric acid.....	74.4	10.0

4. EXPERIMENTAL WORK.

A. Materials Employed.

(1) *Basic Lead Carbonate.* A high grade of commercial white lead was employed, which upon analysis was found to contain lead equivalent to 98 percent of that required by the formula $2PbCO_3.Pb(OH)_2$. It was practically free from other heavy metals. For commercial purposes it is believed that there should be no difficulty in obtaining lead carbonate of adequate purity, since most of the lead used for corroding is nearly free from other metals. No experiments were conducted to determine the effects of probable impurities in the lead carbonate or the lead anodes, although it was observed (as has been found in electrolytic lead refining) that most of the impurities present remain as an insoluble sludge.

(2) *Hydrofluoric Acid.* Some of the experiments were conducted with C. P. hydrofluoric acid and some with commercial acid, principally the 48 percent acid. No appreciable difference in the deposits was caused by the presence of the common impurities in commercial hydrofluoric acid. The principal impurity found was sulphuric acid, present in amounts up to 4 percent. When using such acid, a corresponding amount of the lead is precipitated as insoluble lead sulphate from which the clear solution may be readily decanted. Upon inquiries it was learned from manufacturers that one redistillation would not entirely remove the sulphuric acid. It therefore appears that the cheapest and most convenient way to remove it for this purpose is to use a sufficient excess of lead carbonate to precipitate the sulphuric acid.

(3) *Boric Acid.* The boric acid of commerce was found to be of high purity and entirely satisfactory for this purpose. In general it was found preferable to use crystallized rather than the powdered boric acid, since the latter may react violently with the hydrofluoric acid.

(4) *Glue.* No attempt was made to determine the exact chemical or physical properties of the glue used as an addition agent. Comparison of a number of samples of glue and of gelatin showed that their effects under similar conditions were nearly identical. It is probable that any differences in the quality of the glue will affect the rate at which it is consumed (or at least loses its beneficial action) rather than its specific effect when added. Further investigation will be required to obtain definite data upon this point.

(5) *Lead Anodes.* The lead sheets used for this purpose were secured as "corroding lead," and had the following composition:[13]

	Percent
Copper	0.06
Iron	trace
Nickel	trace
Lead (by diff.)	99.94

It is probable that lead of satisfactory purity may be obtained either as "corroding lead" or as "electrolytic lead." The chief objection to the presence of appreciable amounts of impurities in the lead is the formation of sludge and the consequent increased tendency to form trees upon the deposits.

B. Methods Used.

(1) *Preparation of Solutions.* The solutions are best prepared upon a large scale by placing the requisite amount of hydrofluoric acid in a lead-lined tank and adding the boric acid slowly to it. As the solution becomes very warm during this process, it should be diluted somewhat and allowed to cool before the addition of the lead carbonate. The solution should be stirred while the lead carbonate is added slowly either in the solid form, or preferably in the form of a thick cream. Altogether, however,

[13] Analysis conducted by Mr. J. A. Scherrer, of this Bureau.

no more water should be used than will be required to produce the final volume desired. With some care and experience, the effervescence may be controlled so as not to be excessive. After action has ceased, any lead sulphate is allowed to settle, after which the solution may be siphoned into the plating tank, which should be lined with asphalt. The desired amount of glue (previously dissolved in warm water) is then added and the solution is diluted to the desired volume or specific gravity. In preparing solutions for laboratory experiments the same procedure was followed, except that hard rubber battery jars were employed.

(2) *Analysis of Solutions.* No difficulty was experienced in the determination of lead in such solutions by the usual gravimetric method. This may be conducted by diluting the sample (*e. g.*, 10 cc.) to 100 cc., heating the solution, and adding an excess of dilute sulphuric acid. The lead sulphate may then be filtered out on a Gooch crucible, washed with dilute sulphuric acid, and dried and weighed. Satisfactory results were also obtained volumetrically by precipitating the lead with dichromate, and using the iodide-thiosulphate titration to determine the chromate present in the filtrate. All of these analyses may be conducted in glass, upon which solutions of fluoboric acid have very little effect.

Efforts to determine free fluoboric acid or excess boric acid in such solutions yielded only very unsatisfactory results, owing to the tendency of the fluoboric acid to dissociate or hydrolyze upon dilution or even upon standing. A large number of experiments were conducted in the attempt to develop a method of titration which would yield approximate values, but it was found impossible to obtain even reproducible results. This experience confirms the observations of Abegg, Fox and Herz[14] who concluded that upon mixing hydrofluoric and boric acids an instantaneous reversible reaction occurs, followed by one or possibly several slow irreversible reactions, the end products of which they were unable to determine with certainty. Under such circumstances it is not surprising that inaccurate and erratic results are obtained by titration.

For control of such solutions the lead determination may be employed, with the realization that any increase in lead content

[14] Zeit. Anorg. Chem. (1903), **35**, 129-147.

during operation must be accompanied by a corresponding decrease in free fluoboric acid. Fortunately the solutions do not change rapidly and good deposits can be obtained over a wide range in composition, so that accurate control work is not so essential as in some plating operations.

(3) *Resistivity of Solutions.* The resistivity of the solutions was measured in a simple cell of burette form, with two vertical electrodes, about 1 cm. square, and 15 cm. apart. The cell constant was determined with 0.2 normal KCl. Measurements were made with a 110-volt 60-cycle alternating current, using a Leeds & Northrup A. C. galvanometer. The temperature was maintained at approximately 25° C. (77° F.). No attempt at precision was made in these measurements, since, as previously stated, the composition of the solutions could not be determined with any high degree of accuracy. The results are therefore only of relative value.

(4) *Single Potentials.* Measurements of the potentials of lead in some of these solutions were made, from which the approximate lead ion concentrations of these solutions could be calculated. This information is of interest in determining whether a decrease in lead ion concentration reduces the size of crystals in the deposited lead.

Measurements of the single potentials at 25° C. against a normal calomel electrode were made by Dr. L. D. Hammond, of this Bureau, using a Leeds & Northrup potentiometer and galvanometer. For the reasons previously stated no attempt at precision was made. The values in Table I are expressed in terms of the normal hydrogen electrode, using the value +0.286 V. for the normal calomel electrode. For purposes of comparison the single potential of lead in a normal lead nitrate solution was measured, giving the value —0.156 V.; as compared with the values —0.148 V. and —0.151 V. quoted by Foerster.[15] These values, while by no means coincident, are in sufficiently close agreement to indicate that the relative values obtained in fluoborate solutions illustrate the direction and approximate magnitude of the effects studied. It should be noted that a decrease in the algebraic value of the lead potential (*i. e.*, an increase in the numeri-

[15] Electrochemie Wässeriger Lösungen, 2d Edition, pp. 150 and 152.

cal value) corresponds to a decrease in lead ion concentration, equivalent to a change of one power of ten for each change of 0.029 V. Thus, if (as stated by Foerster) the potential of lead in a solution normal in lead ion concentration is —0.120 V., a potential of —0.149 V. would correspond to a tenth normal lead ion concentration.

A few attempts were made to measure the potential of iron in these solutions, but no concordant results were obtained. Such values would be of interest in determining whether, and if so at what rate, lead would be precipitated upon iron by immersion. It is at least interesting to note that the approximate results obtained in lead fluoborate solutions indicate a very slight difference in potential between the lead and iron; while in fluosilicate solutions an appreciable difference was observed. This behavior is in accordance with the fact previously noted, that lead can be satisfactorily plated directly upon steel from fluoborate but not from fluosilicate solutions.

(5) *Operation of Experimental Baths.* The small-scale experiments were mostly conducted in rectangular glass vessels, each containing about 1,200 cc. of solution. Six such solutions of varying composition were usually operated in series with a copper coulometer, from which the current efficiencies could be calculated. Most of the deposits were produced upon cathodes of cold rolled steel, 5 by 10 cm. (2 inches by 4 inches) *i. e.*, with a total area of 1 sq. dm. They were cleaned and pickled in the usual way. Experiments have shown that when heavy deposits of lead are desired, and especially when they are subsequently required to withstand elevated temperatures, pickling the steel should be avoided, since it tends to cause blisters upon heating. For general use, sand blasting is therefore recommended. Since the sand blast available at the Bureau was not adapted to the treatment of thin sheet metal, the cathodes used in these experiments were cleaned by pickling. By this method a uniform surface was obtained. Comparative tests made upon sand-blasted and pickled specimens showed that there was no essential difference in the structure of the lead deposits produced upon them. Experiments were conducted to determine the structure of thin deposits (0.075 mm. or 0.003 inch) and of relatively heavy deposits (0.254 mm.

or 0.01 inch) produced under the following range of conditions, selected after preliminary experiments:

(a) Lead—From *N* to 2 *N*.

(b) Total fluoboric acid—From *N* to 3 *N*.

(c) Excess boric acid—From 0 to 0.5 *N*.

(d) Glue—From 0.2 to 1.0 g/L.

(e) Current density—From 1 to 3 amp./sq. dm. (9.3 to 27.9 amp./sq. ft.).

(f) Temperature—25° and 50° C. (77° and 122° F.).

(g) Agitation—Still solutions, mechanical agitation, and air agitation.

The current density and voltage required were noted in each case. Attempts to determine the approximate polarization by reading the residual voltage immediately after opening the circuit were unsuccessful, as the polarization in all cases was so small that no definite readings could be secured.

(6) *Permeability of Deposits.* As the chief value of lead plating for either military or commercial purposes is for protection of steel or other underlying metals, the permeability of the lead deposits is of great importance, especially since, unlike zinc coatings, the lead does not furnish intrinsic protection to the steel if the latter is exposed. A very rapid test for porosity may be made by a modification of the well-known ferroxyl test. The samples to be tested are dipped into, or otherwise subjected to, the action of a solution containing about 20 g/L (2.7 oz./gal.) of sulphuric acid, and 10 g/L (1.3 oz./gal.) of potassium ferricyanide. Bright blue spots or stains will appear within a minute wherever the steel is exposed.

In the application of this test, especially under factory conditions, some difficulty was experienced, owing to the presence of superficial particles of iron or iron oxide, derived either from the solutions or from dust in the air. To remove these it was found desirable to wash the surface first with dilute sulphuric acid of equivalent strength but containing no ferricyanide; in order to remove most of the iron particles before the ferricyanide treatment. It is recognized that this test is somewhat too delicate for commercial work but it is at least of value in that if no

definite blue spots are obtained, the coating is certainly impervious.

As preliminary investigation has shown that deposits over 0.25 mm. (0.010 inch) are almost invariably impervious, no permeability tests were made on the heavy deposits. A few tests of lead plated steel in the salt spray yielded results in accord with the ferricyanide test.

TABLE I.

Composition and Properties of Solutions Used.

Soln. No.	Composition				Specific Gravity	Resistivity ohm—cm.	Single Potentials Volts Pb: Soln.
	Total Pb Normality	Total HBF_4 Normality	Excess H_3BO_3* Normality	Glue g/L			
1	1.0	1.0	...	...	1.16	9.2	—0.146
2	1.0	1.5	...	...	1.17	4.5	—0.167
3	1.5	1.5	...	...	1.23	6.8	—0.143
4	1.5	2.25	...	...	1.25	3.5	—0.168
5	2.0	2.0	...	...	1.32	5.6	—0.145
6	2.0	3.0	...	...	1.34	3.1	—0.173
1*B*	1.0	1.0	...	0.2	...	...	....
2*B*	1.0	1.5	...	0.2	...	...	....
3*B*	1.5	1.5	...	0.2	...	...	....
4*B*	1.5	2.25	...	0.2	...	...	....
5*B*	2.0	2.0	...	0.2	...	...	....
6*B*	2.0	3.0	...	0.2	...	...	....
1*C*	1.0	1.0	...	1.0	...	...	....
6*C*	2.0	3.0	...	1.0	...	...	....
3*D*	1.5	1.5	0.5	...	...	...	....
4*D*	1.5	2.25	0.5	...	...	...	....

* Considering H_3BO_3 as monobasic.

C. Results of Experiments.

Including the preliminary experiments, several hundred lead deposits were prepared, the data concerning many of which are of little permanent value. Most of the information obtained in the recent experiments is summarized in Tables I and II, in which are given respectively the data regarding (1) the composition and properties of the solutions and (2) the character of the lead deposits. No actual data for the current efficiencies are given

since in every case the cathode efficiency was over 99 percent, and the anode efficiencies were frequently over 100 percent. This apparent excessive anode efficiency is due principally to the loss of impurities or solid particles and not to actual solution of the lead, since even after prolonged operation there was but a very slight increase in the lead content of the solutions.

TABLE II.

Character of Lead Deposits.

A. Thin Deposits. 0.075 mm. or 0.003 in.

Soln. (of Table I)	Current Density amp./sq. dm.	Temp. °C.	Treeing on Edges	Crystal Structure	Porosity
1	1	25	*B*	*D*	*D*
1	3	25	*C*	*D*	*B*
2	1	25	*B*	*D*	*D*
2	3	25	*C*	*A*	*B*
3	1	25	*B*	*D*	..
3	3	25	*C*	*A*	..
4	1	25	*B*	*D*	..
4	3	25	*C*	*A*	..
5	1	25	*B*	*C*	*C*
5	3	25	*B*	*A*	*B*
6	1	25	*A*	*C*	*A*
6	3	25	*B*	*A*	*B*
1*B*	1	25	*A*	*A*	*A*
1*B*	3	25	*C*	*A*	*C*
2*B*	1	25	*A*	*A*	*B*
2*B*	3	25	*B*	*B*	*C*
3*B*	1	25	*A*	*B*	..
3*B*	3	25	*B*	*A*	..
4*B*	1	25	*A*	*B*	..
4*B*	3	25	*A*	*B*	..
5*B*	1	25	*A*	*C*	*A*
5*B*	3	25	*A*	*A*	*B*
6*B*	1	25	*A*	*B*	*A*
6*B*	3	25	*A*	*A*	*A*
5	3	50	*B*	*B*	*B*
5*B*	3	50	*A*	*A*	*A*
3*D*	3	25	*A*	*A*	..
4*D*	3	25	*B*	*B*	..

TABLE II (Continued).

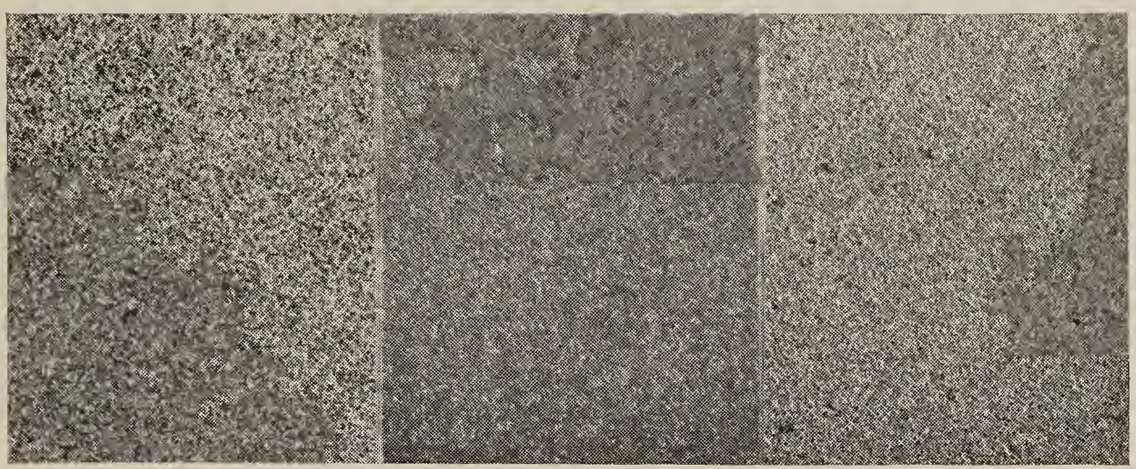
B. Thick Deposits. 0.25 mm. or 0.010 in.

Soln. (of Table I)	Current Density amp./sq. dm.	Temp. °C.	Treeing on Edges	Crystal Structure
1	1	25	*D*	*D*
1	3	25	*D*	*C*
2	1	25	*D*	*D*
2	3	25	*D*	*C*
3	1	25	*D*	*D*
3	3	25	*D*	*C*
4	1	25	*D*	*D*
4	3	25	*D*	*C*
5	1	25	*C*	*D*
5	3	25	*D*	*D*
6	1	25	*B*	*D*
6	3	25	*D*	*B*
1*B*	1	25	*A*	*B*
1*B*	3	25	*D*	*C*
2*B*	1	25	*A*	*C*
2*B*	3	25	*C*	*C*
3*B*	1	25	*A*	*D*
3*B*	3	25	*B*	*C*
4*B*	1	25	*A*	*D*
4*B*	3	25	*B*	*C*
5*B*	1	25	*A*	*D*
5*B*	3	25	*A*	*C*
6*B*	1	25	*A*	*D*
6*B*	3	25	*A*	*B*
1*B**	3	25	*B*	*A*
1*B*	3	50	*B*	*C*
1*C*	3	25	*D*	*B*
6*C*	3	25	*A*	*C*

* Agitated mechanically.

The general appearance and structure of the various types of deposits are illustrated in the photographs (Figs. 1 to 11).

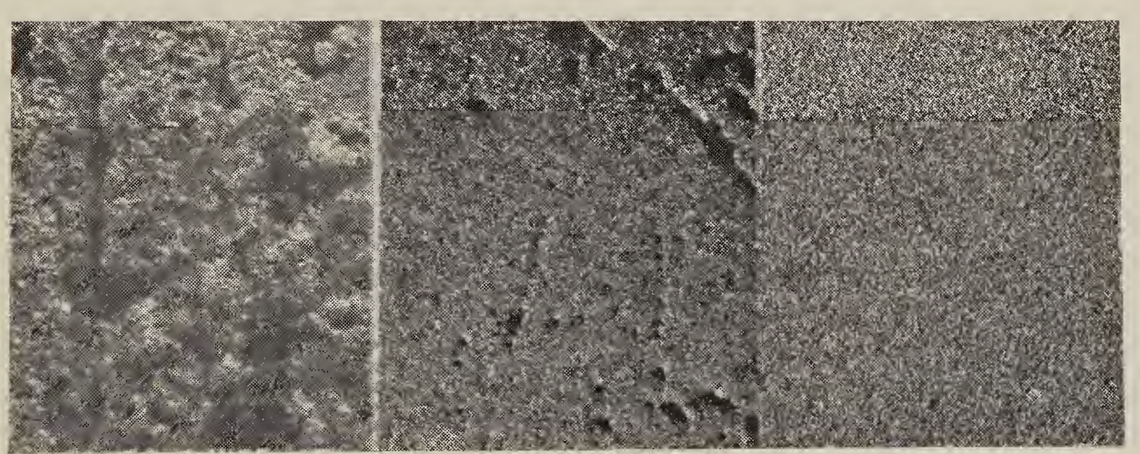
The letters used in Table II have approximately the following significance, illustrated by the photographs referred to. In each case *A* represents the best quality. The ratings given are based upon the opinions of three independent observers.



a b c

FIG. 1.

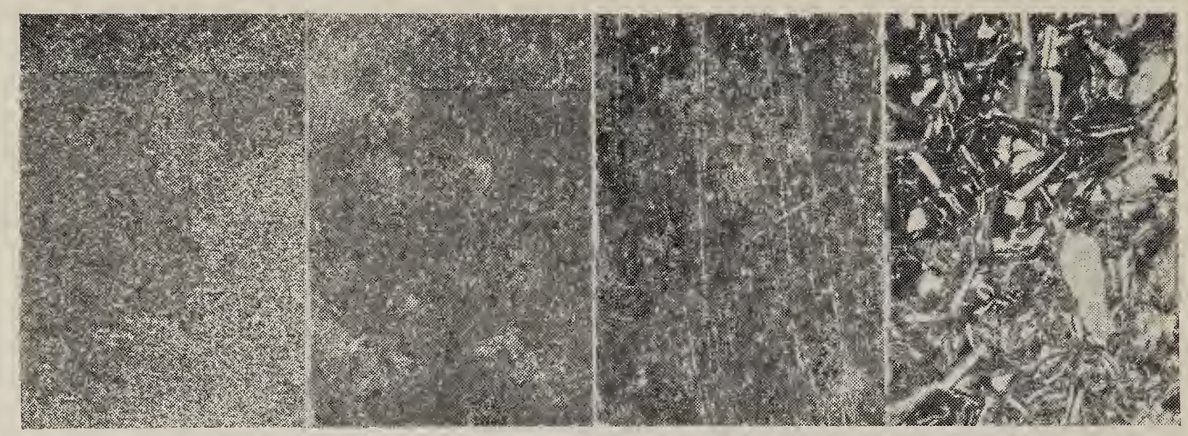
20 diam.
Copper Deposits.
Effect of Current Density.
a 0.2 amp./sq. dm. (1.9 amp./sq. ft.)
b 2 " " " (19 " " ")
c 3 " " " (28 " " ")



a b c

FIG. 2.

20 diam.
Zinc Deposits.
Effect of Current Density.
a 0.2 amp./sq. dm. (1.9 amp./sq. ft.)
b 2 " " " (19 " " ")
c 3 " " " (28 " " ")

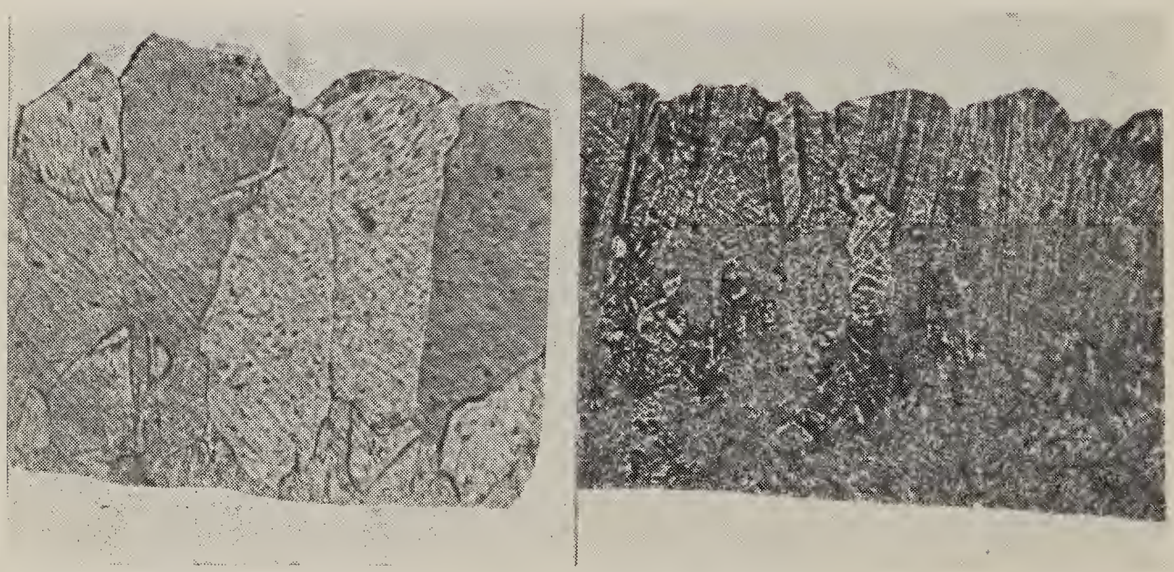


a b c d

FIG. 3.

20 diam.
Nickel Deposits.
Effect of Current Density.

a 0.2 amp./sq. dm. (1.9 amp./sq. ft.)
b 1 " " " (9.3 " " ")
c 2 " " " (19 " " ")
d 3 " " " (28 " " ")

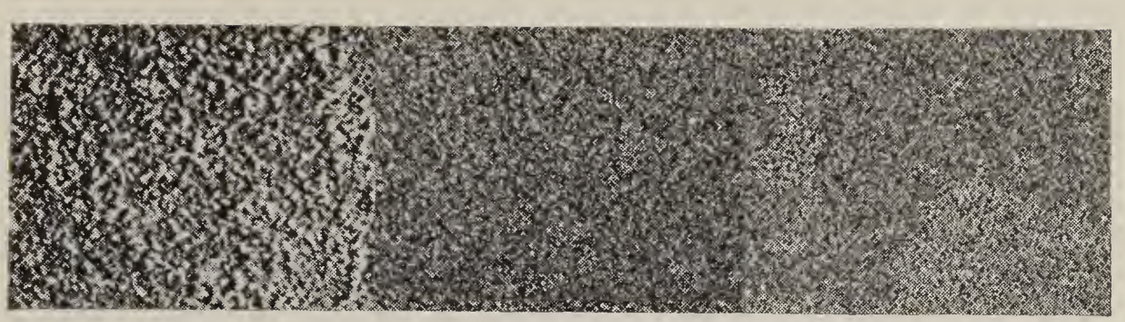


a b

FIG. 4.

300 diam.
Copper Deposits.
Effect of Current Density.

a 4 amp./sq. dm. (37 amp./sq. ft.)
b 10 " " " (93 " " ")



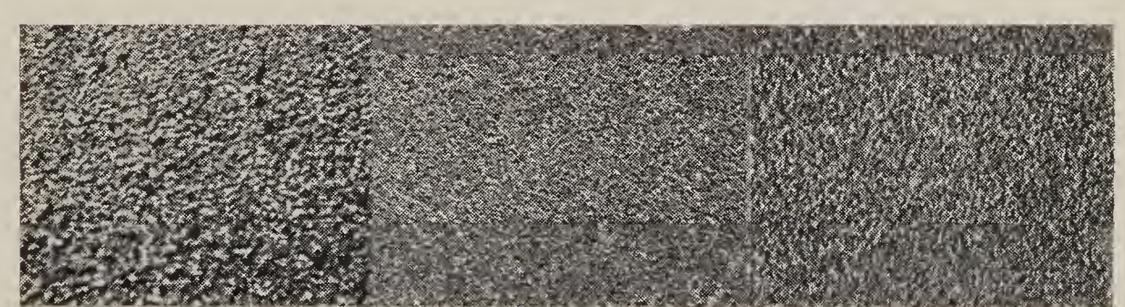
a b c

d e f

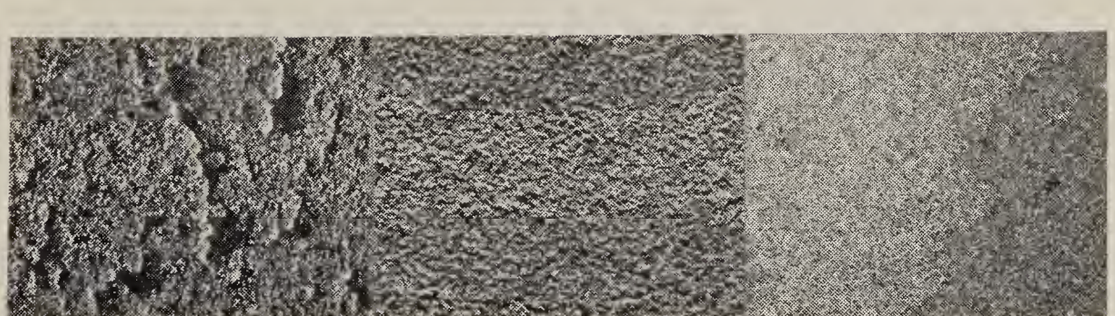
FIG. 5.

20 diam.
Copper Deposits.
Effect of Agitation.

a Still solutions 0.5 *N*
b Air agitation 0.5 *N*
c Mechanical agitation 0.5 *N*
d Still solution 2 *N*
e Air agitation 2 *N*
f Mechanical agitation 2 *N*



a b c



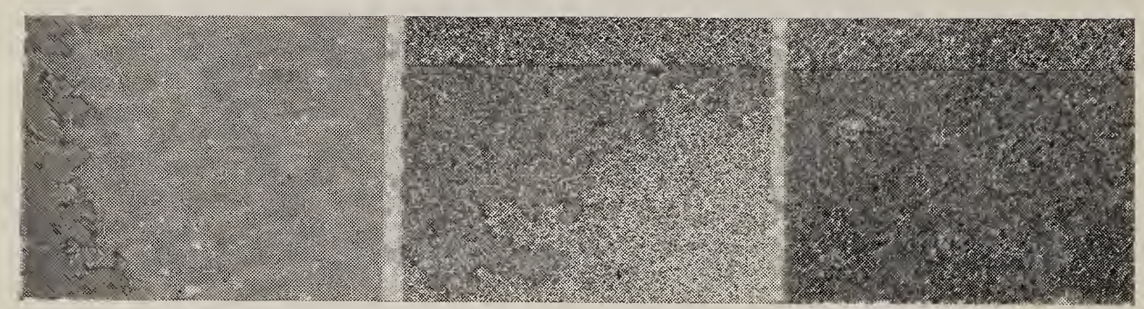
d e f

FIG. 6.

20 diam.
Zinc Deposits.
Effect of Agitation.

a Still solutions 0.5 *N*
b Air agitation 0.5 *N*
c Mechanical agitation 0.5 *N*
d Still solution 2 *N*
e Air agitation 2 *N*
f Mechanical agitation 2 *N*

a b c

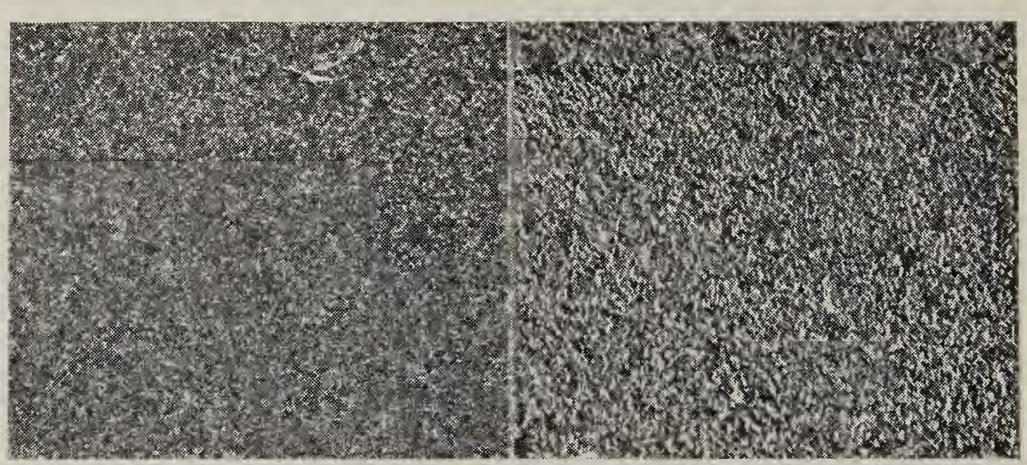


d e f

FIG. 7.

20 diam.
Nickel Deposits.
Effect of Agitation.

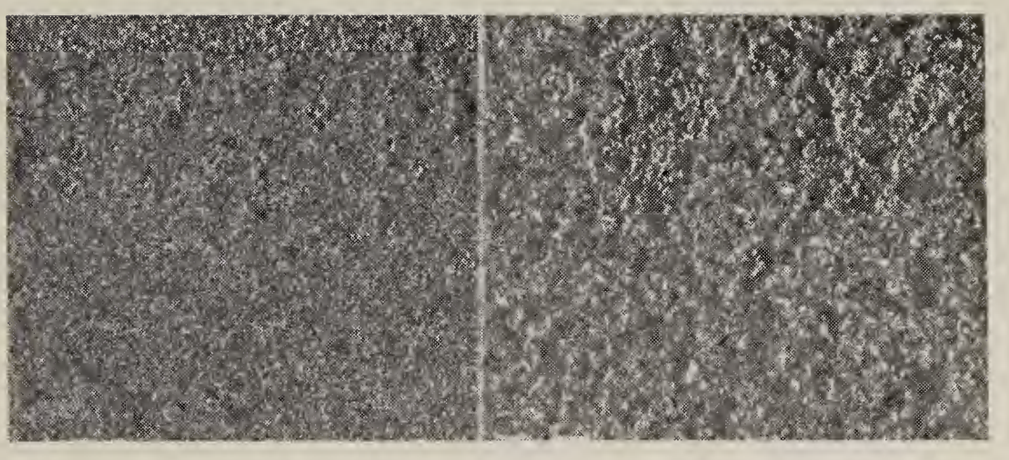
a Still solutions 0.5 *N*
b Air agitation 0.5 *N*
c Mechanical agitation 0.5 *N*
d Still solution 2 *N*
e Air agitation 2 *N*
f Mechanical agitation 2 *N*



a b

FIG. 8.

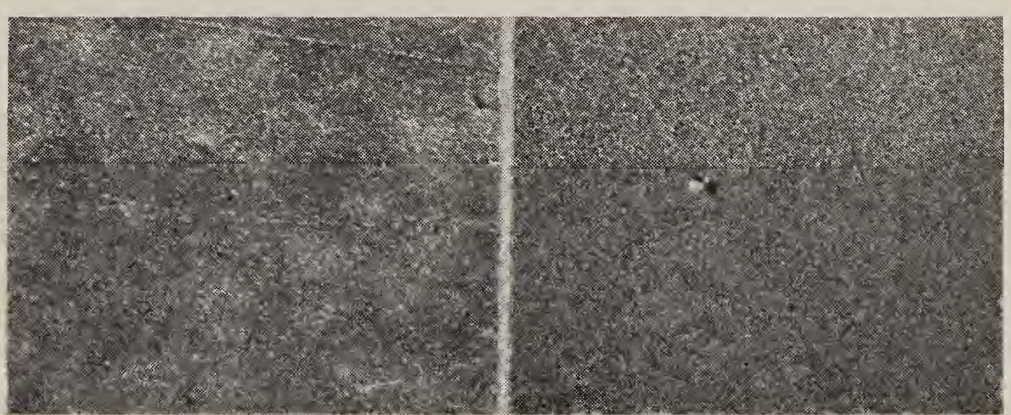
Copper Deposits.
Effect of Temperature.
a 25° C. (77° F.).
b 65° C. (149° F.).



a b

FIG. 9.

20 diam.
Zinc Deposits.
Effect of Temperature.
a 25° C. (77° F.).
b 65° C. (149° F.).



a b

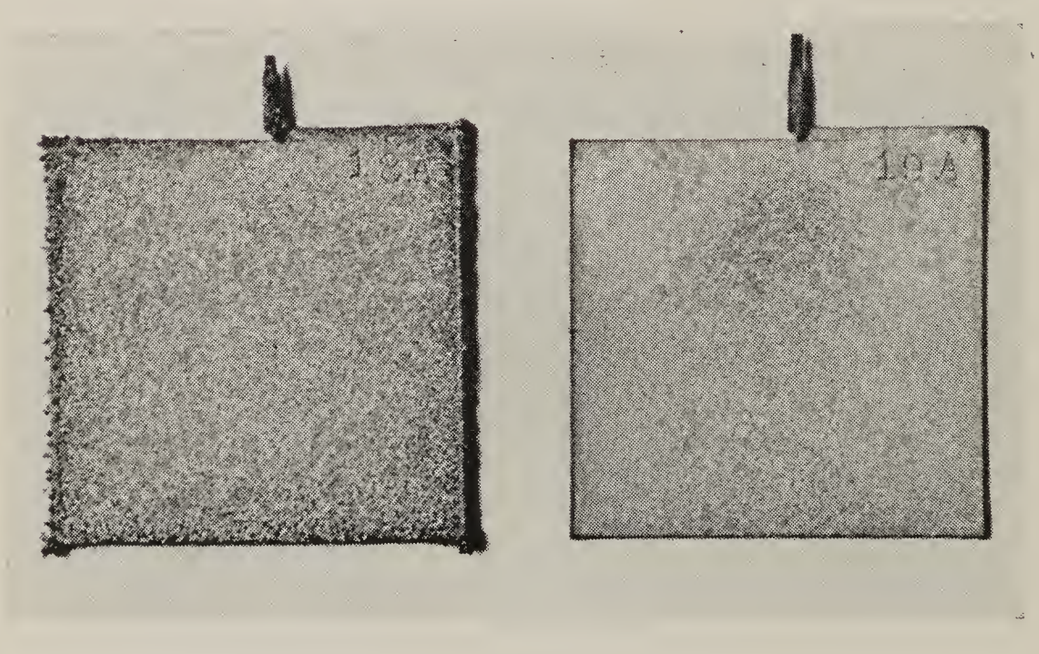
FIG. 10.

20 diam.
Nickel Deposits.
Effect of Temperature.
a 25° C. (77° F.).
b 65° C. (149° F.).

a b

FIG. 11.

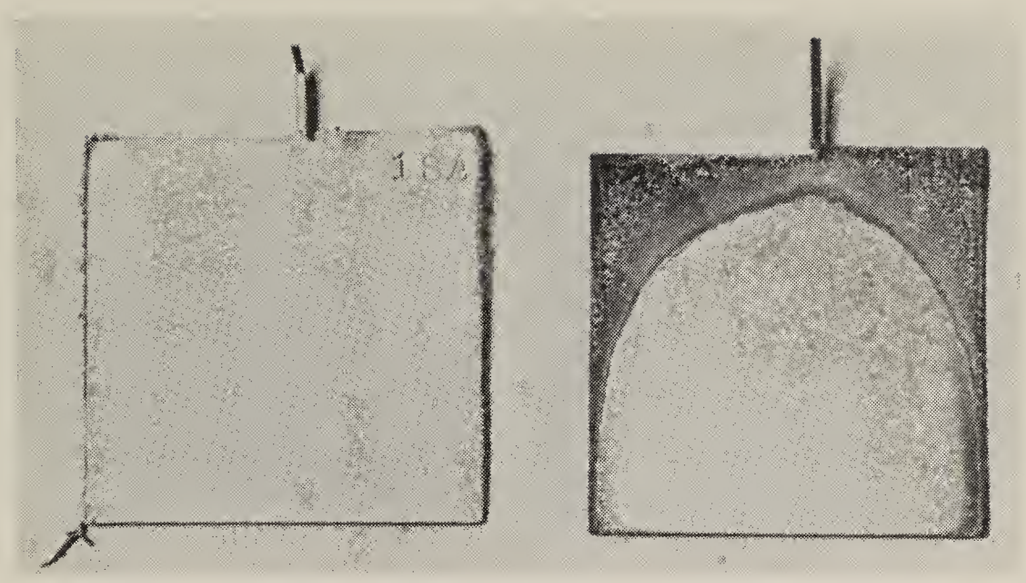
300 diam.
Copper Deposits.
Effect of Temperature.
a 25° C. (77° F.).
b 40° C. (104° F.).



a b

FIG. 12.

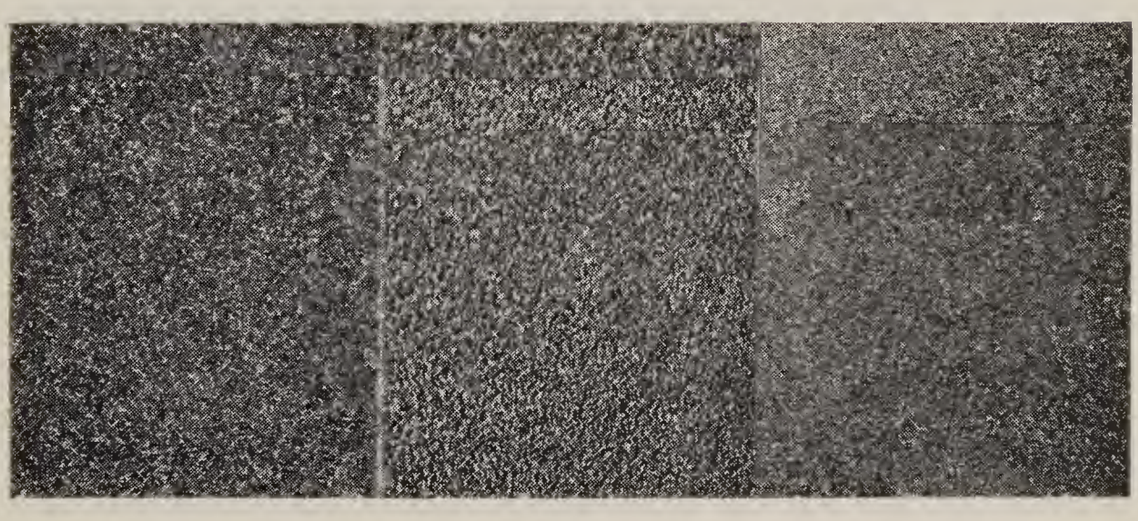
¾ size.
Copper Deposits.
Effect of Conductivity Upon Treeing.
a Copper sulphate *N*, Sulphuric acid 0.1 *N*
b Copper sulphate *N*, Sulphuric acid 1.1 *N*



a b

FIG. 13.

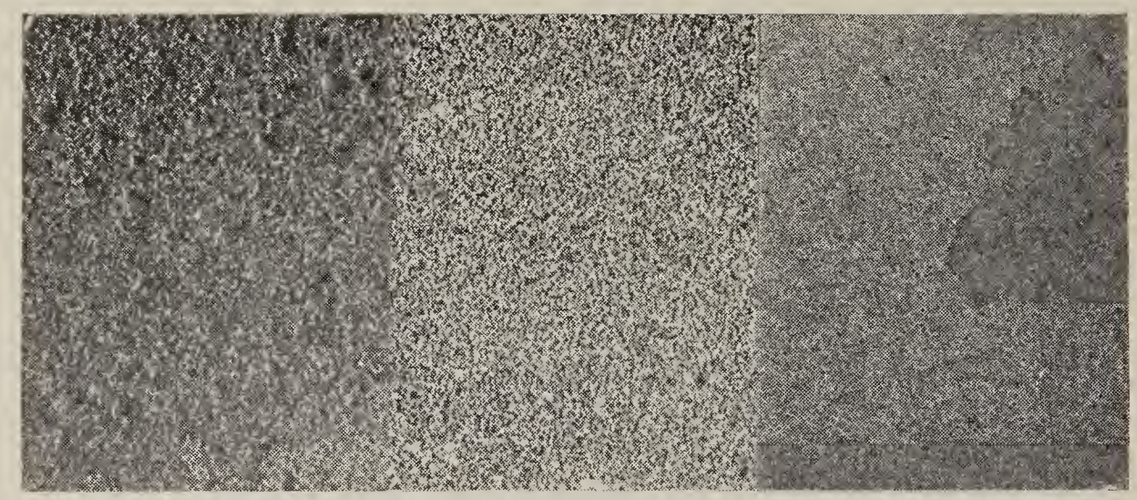
¾ size.
Zinc Deposits.
Effect of Conductivity Upon Treeing.
a Zinc sulphate *N*, Boric acid 0.1 *N*
b Zinc sulphate *N*, Boric acid 0.1 *N*, Sodium chloride *N*



a b c

FIG. 14.

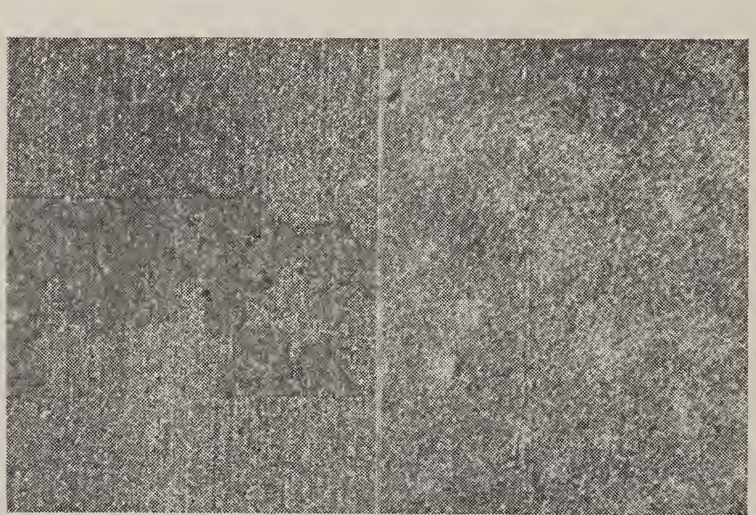
20 diam.
Copper Deposits.
Effect of Metal Ion Concentration.
a "Standard" solution.
b " " + *N* Sodium sulphate.
c Cyanide solution.



a b c

FIG. 15.

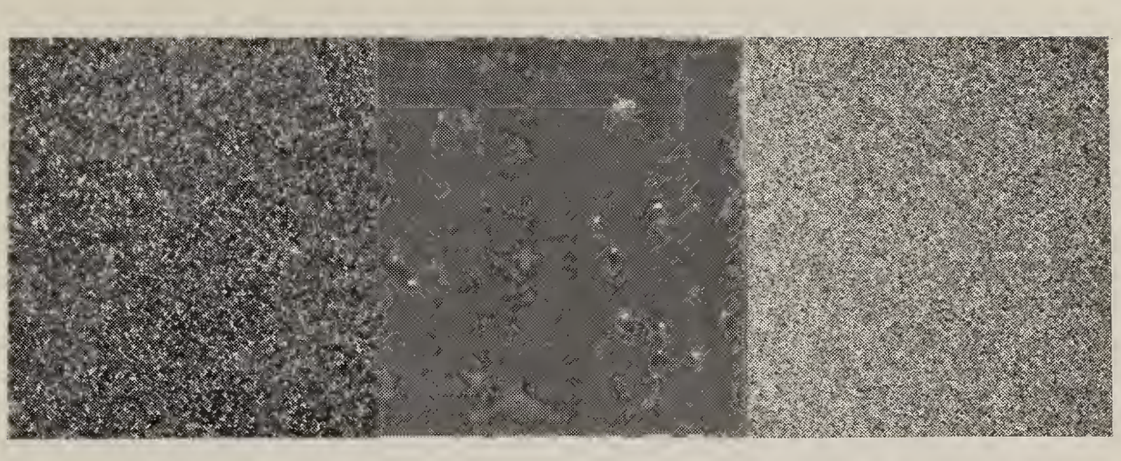
20 diam.
Zinc Deposits.
Effect of Metal Ion Concentration.
a "Standard" solution.
b " " + *N* Sodium sulphate.
c Cyanide solution.



a b

FIG. 16.

20 diam.
Nickel Deposits.
Effect of Metal Ion Concentration.
a "Standard" solution.
b " " + *N* Sodium sulphate.



a b c

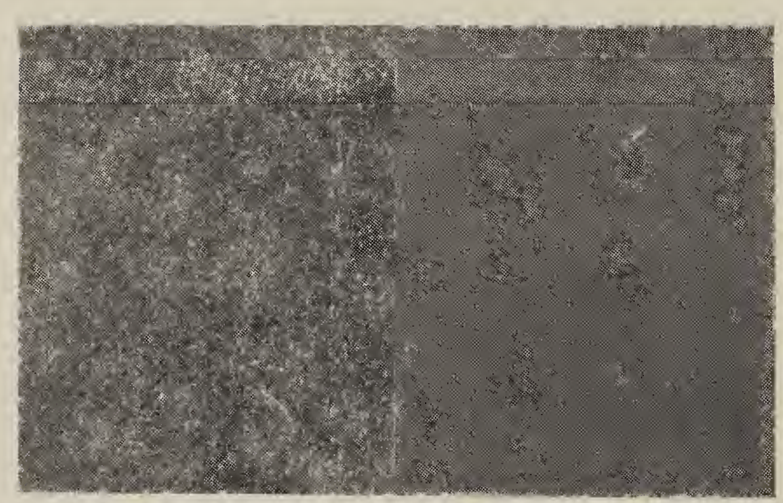
FIG. 17.

20 diam.
Copper Deposits.
Effect of "Addition Agents."
a "Standard" solution.
b " " + glue.
c " " + glucose.

a b c

FIG. 18.

20 diam.
Zinc Deposits.
Effect of "Addition Agents."
a "Standard" solution.
b " " + glue.
c " " + glucose.



a b

FIG. 19.

20 diam.
Nickel Solutions.
Effect of "Addition Agents."
a "Standard" solution.
b " " + glucose.

Treeing on Edges.

Designation	Description	Examples Fig.
A	none	9a
B	slight	8b
C	medium	11a
D	excessive	9a, 11b

Crystalline Structure.

Designation	Description	Examples Fig.
A	very fine	1b or 7a
B	fine	3b or 5a
C	medium	3a or 4b
D	coarse	4a or 7b

Porosity.

A Impervious
B Nearly impervious
C Porous
D Very porous

5. DISCUSSION OF RESULTS.

A. Conclusions.

From the above described and numerous other experiments the following conclusions were reached:

(1) By increasing the concentration of lead it is possible to use higher current densities without causing treeing of deposits. (Figs. 1 and 8.)

(2) An increase in current density increases the tendency to treeing but produces finer grained deposits. (Figs. 4 and 10.) Therefore the highest current density should be employed that will not produce appreciable treeing, since the finer grained deposits are more nearly impervious.

(3) By increasing the concentration of excess (or "free") fluoboric acid slightly finer grained deposits are produced, and there is somewhat less tendency to form trees. (Fig. 2.) This is no doubt due in part to the decrease in lead ion concentration caused by the presence of the excess acid; and in part to the increase in conductivity and consequent decreased tendency to

treeing. From Table I it may be seen that in each case the addition of a 50 percent excess of fluoboric acid caused an (algebraic) decrease of from —0.021 to —0.028 V. If, therefore, as appears probable, the lead ion concentration in the "neutral" fluoborate solutions is slightly more than 0.1 *N*, the addition of fluoboric acid decreases it to between 0.01 and 0.03 *N*. The conductivity of the solutions is doubled by the addition of a 50 percent excess of fluoboric acid.

(4) The presence of an excess of boric acid (above that required to combine with the hydrofluoric acid) has little effect upon the deposits. It is desirable, however, to have an excess of boric acid, since it reduces any tendency to decomposition of the fluoborate and consequent precipitation of lead fluoride sludge.

(5) By the addition of small amounts of glue there is less tendency for the deposit to form trees and the deposits of a given thickness are finer grained and more nearly impervious. (Figs. 3 and 9.). In preliminary experiments it was found that sugar or glucose produces some improvement in the deposits but is not so satisfactory as glue.

(6) By increasing the temperature to 50° C. (122° F.) the deposits become somewhat more coarsely crystalline, and show no marked improvement over deposits produced at ordinary temperature. (Fig. 5.).

(7) Mechanical agitation, *e. g.*, by rotation of the anode or cathode, produces smoother and denser deposits than are obtained in still solutions. (Fig. 6.). This is especially helpful where the deposit must be made from a restricted volume of solution, such as in the inside of a shell or other object. Air agitation has not been found satisfactory, as it produces spongy deposits.

It is interesting to note that in every case these observations and conclusions are in accord with Bancroft's "Axioms,"[16] and furnish an excellent illustration of the value of these axioms as a guide for research in this field.

In view of the very marked effect of colloids and reducing agents in producing finer crystals and especially in reducing the tendency to treeing, a few experiments were carried out with lead

[16] Trans. Amer. Electrochem. Soc. (1904), **6**, 27; (1913), **23**, 266; (1919), **36**, 215.

acetate solutions, such as were employed by Glaser.[17] Betts[18] stated that he was unable to obtain in lead acetate solutions good deposits of lead even after the addition of reducing substances such as pyrogallol which had been employed by Glaser. This observation of Betts was confirmed by us when still solutions were employed, but if as suggested (but not clearly stated or emphasized) by Glaser, the solution was agitated, *e. g.*, by rotating the cathode, a smooth, dense deposit of lead was obtained in solutions containing

Lead acetate	550 g/L
Ammonium acetate	140 g/L
Pyrogallol	1 g/L

An almost identical deposit was obtained in the same solution if glue (0.2 g/L) was added in place of the pyrogallol. In acetate solutions containing no organic addition agent very poor spongy deposits were obtained even with rapid agitation.

These observations indicate that although lead fluoborate solutions are intrinsically superior to lead acetate solutions in that they yield good deposits without agitation or the use of addition agents, the effect of either colloidal or reducing substances is qualitatively the same and even more marked in lead acetate than in lead fluoborate solutions.

B. Recommendations.

The following range of conditions is suggested as a guide for commercial work. For any particular class of work certain modifications may be found necessary. By "thin" deposits is meant deposits of 0.125 mm. (0.005 inch) or less; such as would probably suffice for most applications. "Heavy" deposits include those up to 1.25 mm. (0.05 inch) or more in thickness. The difference in the form and structure of light and heavy deposits is shown in Figs. 7 and 11. This difference is significant from its importance in both practical and research work since any attempt to compare the structure of deposits of lead (or any other metal) of very different thickness is likely to yield very erroneous conclusions.

[17] Zeit. Electrochemie (1901), **7**, 365 and 381.

[18] Lead Refining by Electrolysis, p, 11. Wiley and Sons.

(1) *Composition of Solutions.* For thin deposits a satisfactory solution may be prepared in which the lead concentration is "normal," the total fluoboric acid 1.5 normal, and the excess boric acid 0.2 normal. Such a solution would contain:

	g/L	oz./gal.
Basic lead carbonate	129	17.3
100 percent actual hydrofluoric acid	120	16.1
Boric acid	106	14.2
Glue	0.2	0.025

In using commercial materials to prepare such a solution it should be borne in mind that the hydrofluoric acid is never 100 percent HF. Commercial acid is usually furnished in three grades, designated respectively as 30 percent, 48 to 52 percent, and 60 percent acid. For the preparation of plating solutions it will usually be found most convenient to use the 48-52 percent acid, which may for practical purposes be considered at 50 percent HF. In this case, it is necessary to use twice the above specified amount, that is, 240 grams per liter or 32 ounces per gallon. The commercial hydrofluoric acid always contains some sulphuric acid, in some cases as much as 4 percent. Since the sulphuric acid will precipitate an equivalent amount of the lead as lead sulphate it is necessary to use a sufficient excess of lead carbonate to combine with the sulphuric acid present. This may be determined by analysis in any given sample of hydrofluoric acid, but on the average it may be assumed to contain 2 percent of sulphuric acid which for 240 grams per liter of 50 percent hydrofluoric acid, is equivalent to about 13 grams per liter of lead carbonate. The amount of the latter should therefore be increased by about 10 percent. The actual materials to be used would then be:

	g/L	oz./gal.	Practically oz./gal.
Basic lead carbonate	142	19	20
50 percent hydrofluoric acid	240	32.2	32
Boric acid	106	14.2	14
Glue	0.2	0.025	0.025

This solution has a specific gravity of about 1.17 (21° Baumé).

For heavy deposits or for rapid deposition with high current densities or when it is desired to operate without any addition

agents such as glue, stronger solutions should be used, up to twice the above concentrations; in this case the solution would have a specific gravity of about 1.34 (37° Baumé).

Occasional small additions of glue will be required whenever the deposits show any tendency to roughness or treeing. With any given class of work the desired rate of addition of glue can be readily determined. In a few small-scale experiments it was found that over 800 grams of lead could be deposited from 1,200 cc. of fluoborate solution containing about 0.24 g. of glue, before pronounced trees were formed. Even after this period of operation the effect of the glue was noticeable in that the formation of trees was less pronounced than in a solution to which no glue had been added. No effort was made to determine the maximum glue content permissible, but from experiments conducted with solutions containing as much as 1 g/L of glue, it was found that the deposits, especially from dilute solutions, were slightly inferior to those with 0.2 g/L of glue.

(2) *Current Density.* For thin deposits, when using the dilute solution (specific gravity 1.17) a current density as high as 3 amp./sq. dm. (28 amp./sq. ft.) may be used on smooth or regularly shaped articles. For general work, however, it will usually be found most satisfactory to use not over 2 amp./sq. dm. (19 amp./sq. ft.) in the dilute solutions, and not over 3 amp./sq. dm. (28 amp./sq. ft.) in the concentrated solutions (sp. g. 1.34). When heavy deposits are required not over 1 amp./sq. dm. (9 amp./sq. ft.) should be used in the dilute solution, or 2 amp./sq. dm. (19 amp./sq. ft.) in the concentrated solution, as the tendency to form trees on heavy deposits is greater with high current densities. The voltage required for any current density depends upon the solution used and the shape of and distance between the electrodes. In every case it is comparatively low and rarely exceeds 1 volt, since these solutions, especially those containing free acid, are well conducting.

(3) *Agitation.* For most purposes agitation (except occasional stirring to keep the solutions uniform) will not be required. When plating in a restricted volume or on the inside or outside of a cylindrical surface mechanical rotation will be found advantageous.

(4) *Temperature.* There is no advantage in heating the solutions above room temperature; in fact if the current used produces marked heating of the solution, *e. g.,* to above 40° C. (104° F.), it will be an advantage to cool the solutions.

(5) *Preparation of the Surface.* Wherever practicable sand-blasting should be used instead of pickling. This is absolutely essential if the lead deposits are subsequently required to withstand elevated temperatures, *e. g.,* in chemical equipment. Experiments have shown that lead deposits produced from fluoborate solutions upon sand-blasted surfaces may be heated to 250° C. (480° F.) without appreciable blistering or loosening of the deposit. Where pickling in hydrochloric acid or sulphuric acid is used, it should continue for the shortest possible time.

(6) *Permeability.* The exact thickness of lead required to give an impermeable coating when tested with ferricyanide, will depend upon the smoothness and cleanness of the initial surface and upon the structure of the lead deposit. Under favorable conditions, especially from solutions containing glue, a thickness of 0.075 mm. (0.003 inch) is sufficient, but 0.125 mm. (0.005 inch) is recommended. With light coatings a slight burnishing or even scratch brushing will serve to render the coatings less porous. If the lead coatings are to withstand corrosive liquids deposits of 1.25 mm. (0.05 inch) or more may be required.

6. APPLICATIONS OF LEAD PLATING.

With increased familiarity with lead plating, it is probable that there will be new commercial applications. The solutions are very readily operated and produce satisfactory results over a wide range of conditions. They are not so poisonous as many common plating solutions and they are not unduly expensive.

The commercial applications of lead plating must be sought in those fields where it is desirable to protect steel or other metals against certain acids or other corrosive liquids. It should be borne in mind, however, that lead is not suitable for protection against acetic or nitric acids. It is not logically a substitute for zinc coatings, which, while they themselves are more readily attacked, exert protection to the underlying steel even if the latter is exposed. Lead coatings exert no such protection, and must there-

fore be impervious to give lasting protection. In many cases lead plating may prove to be a substitute for tin plate or terne plate, either because of the high price of tin or of possible objection to the presence of the tin which is added to lead in making terne plate.

Among the uses of lead plating which have been suggested, and are being investigated commercially are: (1) The coating

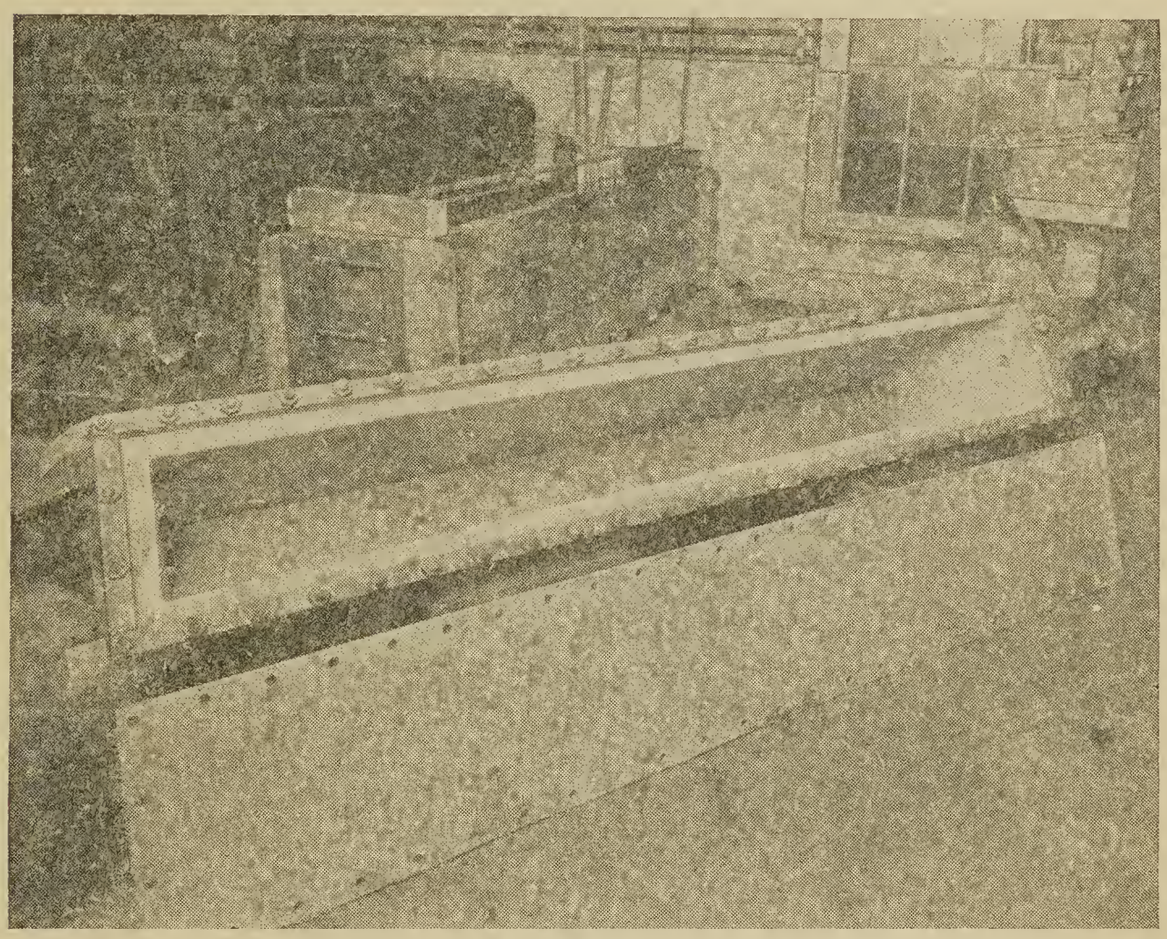


FIG. 12.

of sheet iron to be used in making gas heater flues, in which sulphuric acid may be condensed. The use here necessitates control of the operating temperature to avoid melting of the lead. (2) The lining of copper fire extinguishers of either the soda-sulphuric acid or the carbon tetrachloride type, since under some conditions the copper in these may in time be appreciably attacked. (3) The lining of tanks used to contain brine for refrigeration. (4) The lining of chemical apparatus, especially vacuum stills or other forms to which sheet lead cannot readily be attached.

As an example of the latter use the experiment conducted upon a large catalyzer box for the Edgewood Arsenal is of interest. This box was constructed of steel, electrically welded, and had a volume of about 150 gallons (568 liters) and an inside area of over 50 sq. ft. (5 sq. m.). Using a fluoborate solution similar to that recommended, and slightly less concentrated than the most concentrated solution above suggested (*i. e.*, specific gravity about 1.29 instead of 1.34), lead was deposited for about 50 hours at a current density of 10 amp./sq. ft. (1 amp./sq. dm.). The resulting nearly smooth deposit was about 0.07 inch thick and weighed about 275 lbs. (125 kg.). (Fig. 12.). Owing to the fact that this box was completed just before the signing of the armistice, there was no opportunity to test it in actual service. There is every reason to believe, however, that the deposit would have met the requirements, and that it suggests the possibilities of lead plating upon an even larger scale.

The following factors may be found useful in the practical operation of lead plating:

Atomic weight of lead = 207.2
Specific gravity of lead = 11.3.
1 square foot of lead, 0.001 inch in thickness, weighs 0.954 oz.
1 oz. of lead per sq. ft. = 0.001044 inch
1 lb. of lead per sq. ft. = 0.0167 inch
7.3 ampere hours deposit 1 oz. of lead
1 ampere requires 7 hours and 20 minutes to deposit 1 oz. of lead
117 ampere hours deposit 1 lb. of lead
7.0 ampere hours per sq. ft. deposit 0.001 inch lead

7. SUMMARY.

Lead plating is a practical operation, which can be conducted by any experienced plater with the usual facilities. There is a considerable field for its future application.

In conclusion the authors desire to acknowledge the assistance of various members of the Bureau staff, and especially of Mr. Geo. B. Hogaboom, for information regarding commercial lead plating obtained by him in visits to plating plants.

Bureau of Standards,
Washington, D. C.
July, 1919.

DISCUSSION.

WILLIAM BLUM: Lead plating was one of those industries which was developed as a commercial possibility practically as the result of military need. Towards the end of the war lead plating came into extensive use in connection with gas shells and boosters for gas shells, and for bringing up to weight underweight shells. The paper which we present is an effort to bring together the existing information upon lead plating, including the results of a considerable number of experiments in which we determined the effect simply of the most obvious variables in lead fluoborate solutions.

F. C. MATHERS[1]: I would like to ask, on the point brought out that the fluoborate is better than the fluosilicate, what is the difference in the deposits from these two baths?

WILLIAM BLUM: The differences observed were as follows: In commercial operations, at the time when the subject was first investigated, it was found that the fluosilicate solutions could not be used to deposit directly upon steel, which was always therefore first covered by a preliminary coating of copper; whereas it was found perfectly practicable to deposit lead directly upon steel by means of a fluoborate solution. Subsequent to that time, Mr. Thum developed and patented a method by which the fluosilicate solution could be used directly upon steel, provided the steel was first immersed in a solution of glue. No explanation was offered for this behavior, but it may perhaps be accounted for by the fact that the glue delays the rate of reaction between the steel and the fluosilicate solution. We did not make any extensive investigations of fluosilicate solutions, but from the few we made we found that under similar conditions the deposits from fluosilicate solutions were not so fine grained or impervious as from fluoborate solutions. Incidentally the fluoborate solution is easy to prepare from hydrofluoric and boric acids, whereas the fluosilicate solution involves the use of fluosilicic acid, which is not readily prepared on a small scale.

[1] Asst. Prof. of Chemistry, University of Indiana.

F. C. MATHERS: I wish to bring out some points concerning addition agents in lead baths. The effects of the various possible addition agents in lead baths have never been thoroughly studied. At least no such research has ever been published. Glue gives good results, but I am not convinced that it is the best addition agent. There is a possibility of finding substances that will give better results in these baths. The glue appears to be perfectly satisfactory in the fluoborate bath and fairly satisfactory in the fluosilicate bath, but it does not follow that glue will give a good deposit from another lead bath. An addition agent often loses its value when another salt of the metal is used in the bath. The lead perchlorate bath, which is said to give the best deposit of any lead bath, gives a crystalline rough deposit when glue is used, as is shown by this sample cathode. However, clove oil in the perchlorate bath gives a splendid solid, smooth, finely crystalline deposit, as is shown by this sample. Neither glue nor clove oil gives good deposits from lead acetate, lactate or formate baths, but aloin, in these baths, gives fairly satisfactory cathode deposits which these samples show to be firm and solid, although brittle.

ANSON G. BETTS[2] (*Communicated*): It has been fully known for some fifteen years that lead plating could be readily and successfully carried out, producing an entirely satisfactory and perfectly solid lead coating by the same method as used on a large scale in refining lead electrolytically, and the process has been fully described. In many respects much fuller details than those contained in this contribution have been given by myself, especially in my book "Lead Refining by Electrolysis."[3] That the deposits readily obtained were as good as any electro-deposit, and were perfectly solid as evidenced by specific gravity determinations, was fully pointed out and tabulated as follows. (Glue was used in all solutions.)

Solution	Specific Gravity of Deposits		
Lead Fluosilicate	11 29	11.35	11.36
" Benzyl-sulphonate	11.35	11.37	
" Ethyl-Sulphate	11.27	11.31	
" Fluoborate	11.39		
" Dithionate	11.20		
" Phenol-sulphonate	11.35		

[2] Nyack, N. Y.

[3] "Lead Refining by Electrolysis," John Wiley & Sons, New York, 1908.

That lead fluoborate solutions gave unexcelled deposits was fully known, as well as the fact that the solution was easily made, but the fluosilicate was adopted for refining for reasons of greater economy.

The chief reason for the non-use in lead plating on any considerable scale of my discoveries was much the same as the reason why copper is not electro-plated on any scale, although everyone knows that it is easy to make good copper deposits. That copper-plated steel wire would be very desirable for telephone and telegraph wires is quite obvious, and I believe at one time the Western Union Telegraph Company had a plant for making it. Of course the chief application for lead-plating would be for covering iron and steel. But commercial iron and steel universally contains imperfections in the shape of particles of grit or slag, some of which always show on the surface, and do not take the coating, with the result that there are pin holes in the coatings, at which corrosion starts. Much effort has been given to overcome this, including scratch-brushing and other mechanical treatment. If the authors of this paper or anyone else could devise a way to overcome this very serious trouble, or if steel mills would put out a product as homogeneous as copper wire bars or ingots, they would be really doing something that would lead to great applications of copper-plating, lead-plating, and electro-galvanizing. Otherwise the value of lead-plating is bound to be much restricted, including its use for lining chemical apparatus. It might be noted that the authors avoid any reference to this practical matter, although everyone who has seriously tried electro-plating of iron and steel must be aware of it, and anyone inclined to go ahead commercially at the authors' suggestion that it is all right, would do well to consider it.

The authors state, on page 259, that they secured a "smooth, dense deposit of lead" from a solution of lead acetate containing pyrogallol, which ordinarily deposits merely a loose deposit of spangles or branching crystals, by rapidly rotating the cathode. The inference of this would be, since the paper treats of lead plating, that in this way a good plating deposit is obtained. However, this should not be accepted without proof. Rotation of the cathode can well disturb the crystalline growth to some extent, and make a better looking deposit, but not one necessarily solid,

and it is very doubtful if the authors or anyone else has made solid deposits in this way. Such statements should not be made unless backed up by something definite, with all conditions of the experiment stated. There is a very easy way of making a measurement of solidity, by first making a deposit of some considerable thickness and then taking the specific gravity; and as the authors have cited my patents and are no doubt familiar with my contributions and book on the subject, although ignoring them, and as the specific gravity method was fully used and stated, they are no doubt familiar with it, so they have merely made what I consider a loose statement, and have not taken an easy way of submitting some proof.

Snowdon[4] claimed to have produced solid deposits of lead from the acetate solution without crystal-restraining agents, but also did not give anything definite in the shape of figures for solidity. He used the same device of rapid rotation of cathodes.

WILLIAM BLUM (*Communicated*): As pointed out by Dr. Betts, the preparation and condition of the surface for lead plating are very important if impervious deposits are required. For this reason the authors of this paper have (on page 262) advocated the use of sand blasting, and have also laid stress on the fact that the porosity of the deposit depends directly upon the condition of the surface. In the plating of shells, and of the tank referred to in the paper, sand blasting proved effective. Whether it will prove conveniently applicable must be determined in each particular case.

Although familiar with the experiments upon the density of electro-deposited lead, as described by Dr. Betts in his patent and book (both of which are cited in this paper), the authors made no density measurements, but have depended rather upon the crystal structure and permeability tests as criteria of the protection likely to be afforded by a lead coating. Except insofar as it is an indication of impermeability (which property can be measured directly) the density of the deposited lead is usually not of interest in connection with protective coatings.

[4] Transactions Am. Electrochemical Society (1906), 9, 221.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

DEPRECIATION IN SMALL DRY CELLS WITH AGE.[1]

By A. J. Helfrecht.[2]

Abstract.

The author endeavors to show how closely the method of judging cell deterioration, called "flash test," approaches actual measurements of capacity through discharging the cells. Comparative curves for the different sizes of cells tested are given. From the data gained by this investigation a table has been compiled indicating reasonable depreciation of the four important sizes of small cells.

It is well known that small dry cells undergo a steady depreciation in capacity from the time that they are made, and a knowledge of the rate at which such depreciation takes place has become of great technical importance because of the extensive use to which dry cells are being put, and because of the new and exacting demands which are being placed upon this type of cell in its application to wireless telegraphy and telephony.

The term "shelf deterioration" is employed to designate the decrease in capacity which a dry cell undergoes when not in use. This factor has been touched upon in various papers presented to this Society,[3] and it is the purpose of this paper to present further data on this subject dealing specifically with the smaller sizes of cells.

The actual determination of the capacity of a dry cell involves its discharge, and, therefore, its destruction; hence, to determine the rate of deterioration over a period of time by this method,

[1] Manuscript received July 22, 1919.

[2] Chemical Engineer, C. F. Burgess Laboratories, Madison, Wisconsin.

[3] Transactions of the American Electrochemical Society (1908), 13, 17, J. W. Brown; (1909), 16, 109, F. H. Loveridge; (1910), 17, 341, D. L. Ordway; (1912), 19, 31, W. B. Pritz; (1918), 34, 75, C. A. Gillingham.

tests must be made upon a sufficiently large number of cells of identical construction to give the required information. To avoid the complication and expense involved by such method there is need of a deterioration test which can be made on a single cell without its destruction by the testing method. But no such test has been evolved which gives more than a crude approximation. It is a common but a decidedly misleading practice of numerous battery makers to guarantee their product not to deteriorate more than a specified percentage over a given period of time as judged by the decrease in voltage. The absurdity of this method is shown by the fact that the open circuit voltage of a cell has little relationship to its watt hour capacity. A cell may decrease 50 percent in actual capacity, while its open circuit voltage may decrease not more than 10 percent. A more rational, but still unsatisfactory method which has had some use is to judge the deterioration of a cell by measuring its "flash" or its instantaneous short-circuit current at various intervals. It is known that as a cell deteriorates with age its internal resistance increases and consequently its flash decreases.

The main purpose of this paper is to present results showing how closely this method of judging cell deterioration approaches the method involving the actual measurement of capacity through discharging the cells.

Cells Tested.

Cells of four different sizes representing the range of the flash light cells on the market were employed in this test. Part of these were purchased on the open market of a standard well-known make, and to eliminate the factor of uncertainty as to the time which elapsed from the date of manufacture to the date of purchase, many other cells of these same sizes were made up in our laboratories by trained workmen, using standard materials, such as are used in the regular marketed product, special care being taken to avoid variations in construction between the different cells.

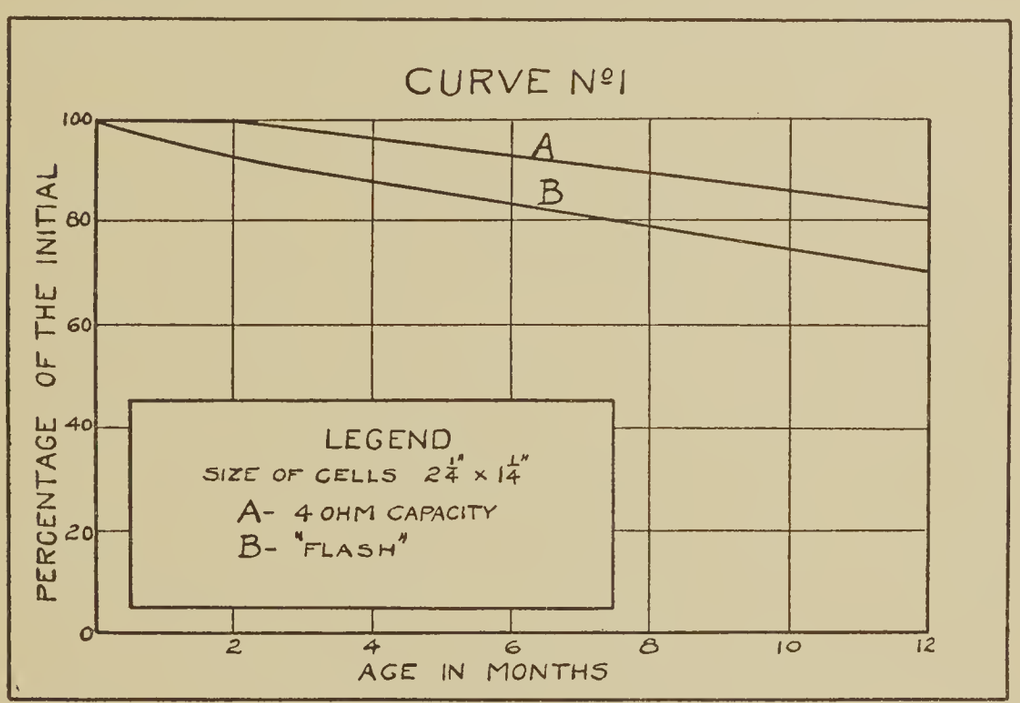
Methods of Testing.

Although standard methods for testing dry cells have been recommended by the American Electrochemical Society,[4] and

[4] Committee on Dry Cell Tests, Tr. Am. Electrochem. Soc. (1912), 21, 275.

although the Bureau of Standards has issued recently a circular[5] on dry cells in which standard tests similar to those advocated by this Society have been recommended, it is unfortunate that manufacturers and large users of dry cells have not yet settled upon uniform testing methods.

In the investigation covered by this paper the "flash" tests were made by short-circuiting each cell through a low-resistance Weston ammeter for as short a period as would indicate the steady maximum deflection of the needle. This test was repeated periodically over a year's time to indicate the deterioration. Since the initial flash differs greatly between individual cells of one



Curve No. 1.

make, the decrease of flash as time passes is expressd not in terms of decrease in amperes but in percentage of the initial flash when the cells are first put on test. This method places the readings as between different batteries on a more common basis.

Capacity tests were made by a method described in detail in "Characteristics of Small Dry Cells," by C. F. Burgess,[6] and designated here as the "4-ohm" test, in which each cell is connected to a 4-ohm coil and discharged eight hours per day until the closed circuit voltage drops to 0.5 volt.

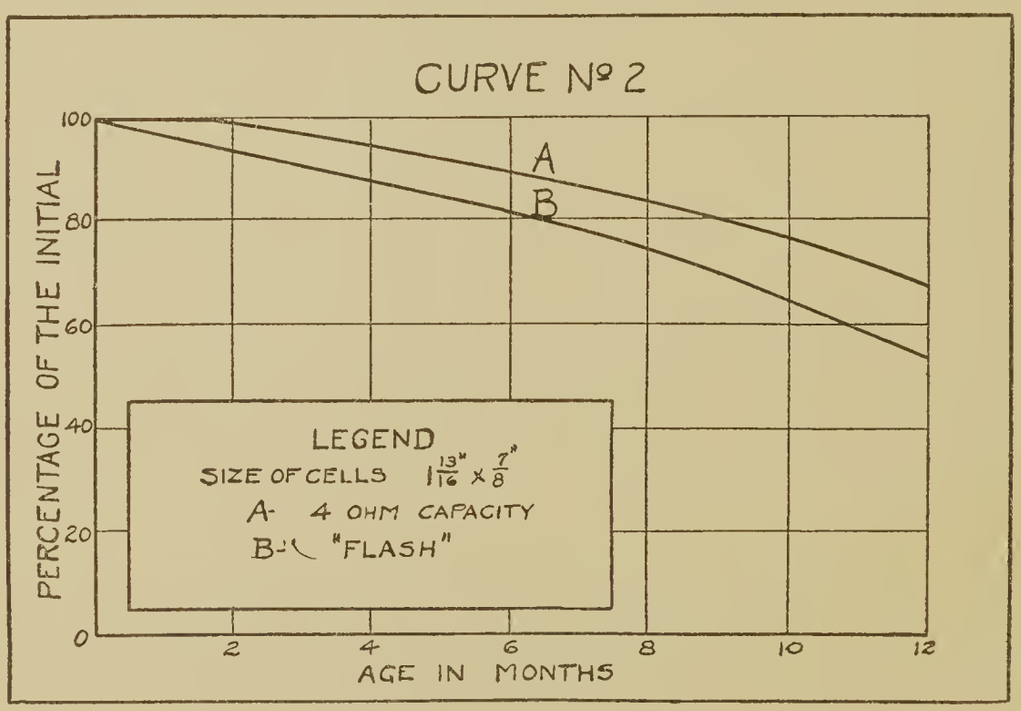
[5] Circular No. 79, Electrical Characteristics and Testing of Dry Cells.
[6] Trans. Am. Electrochem. Soc. (1916), **30**, 257.

The corresponding test recommended by the Bureau of Standards consists in the use of a 2.75-ohm coil and discharging to a similar end point of 0.5 volt.

The "4-ohm" test used for the investigation set forth in this paper was adopted prior to the publication of the bulletin of the Bureau of Standards and to draw a comparison between these two tests a special lot of cells was made up and tested by the two methods.

Results.

In comparing the "4-ohm" test for capacity with the "2.75-ohm" test and expressed in time of service until the cells reached the

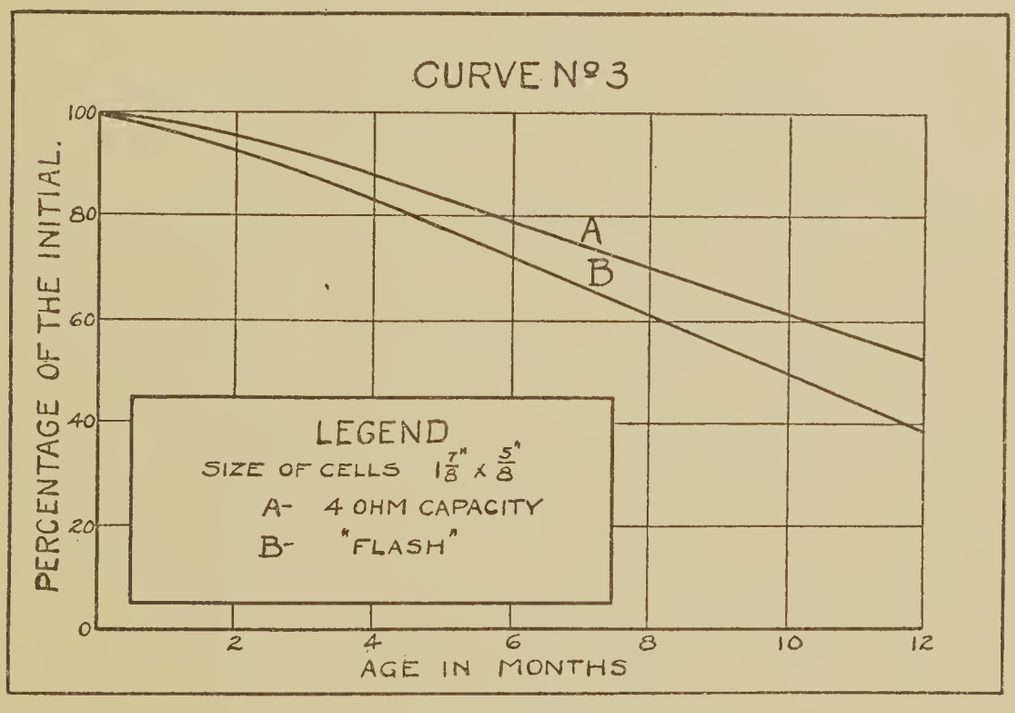


Curve No. 2.

0.5 volt end point, the average of numerous measurements showed that the time of service on the latter test is 60 percent of that of the former with a range between 55 percent and 65 percent. In the accompanying curves, No. 1 shows the depreciation of the most largely used size of flash-light cell, such as is employed in tubular handlamps, 1½ in. (3.2 cm.) diameter and 2¼ in. (5.7 cm.) high. Graph *A* shows the percentage decrease in capacity during a period of one year as determined by the "4-ohm" test. It is to be noted that there is no appreciable depreciation in capacity during the first two months, and during the remaining

ten months the depreciation of about 17 percent follows approximately a straight line. The corresponding graph *B*, derived by the "flash" test indicates an almost linear depreciation from the beginning of the test. The notable deduction from this curve sheet, as well as from the others which follow is, that the depreciation indicated by the "flash" test is decidedly greater than that measured by the actual capacity.

Curves Nos. 2, 3, and 4 represent in a similar way tests on smaller sizes of cells and indicate clearly the variations in performance as the cells decrease in size.

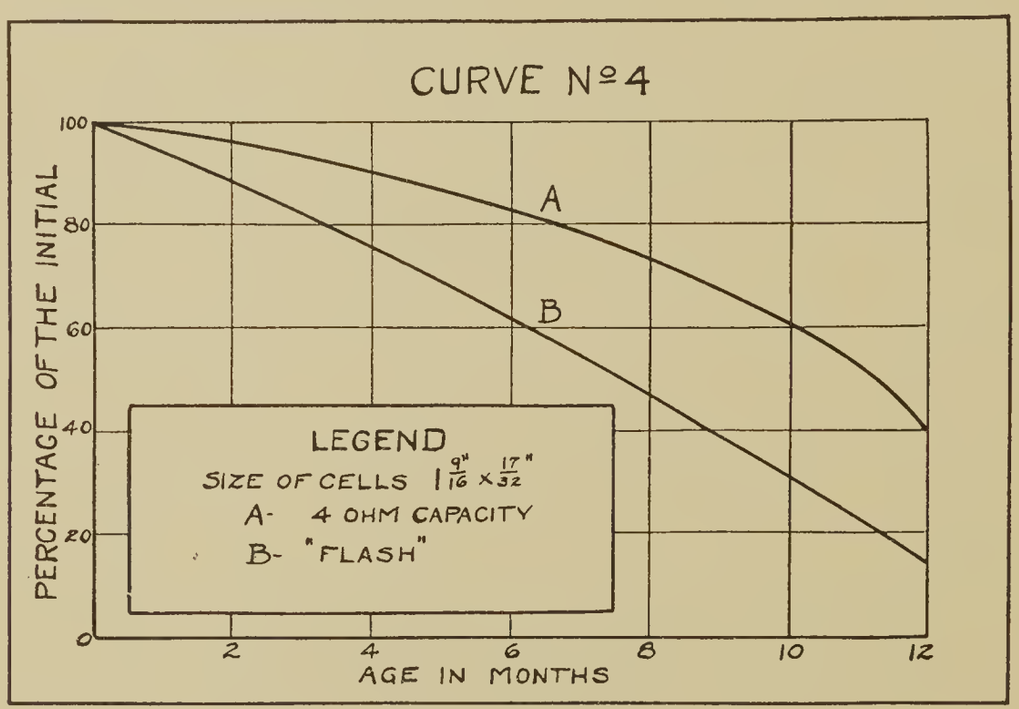


Curve No. 3.

The curves as a whole show that the actual depreciation as measured by the capacity tests is less than the apparent depreciation determined by the flash tests and this departure becomes especially marked in the smallest size of cell. This difference is due to the fact that the "flash" test is influenced mainly by the internal resistance of the cell which slowly increases with age. Since this internal resistance is but small as compared with the resistance through which the cells deliver current when working normally or when placed on capacity test, the increased internal resistance does not necessarily influence to a large degree the ability of the cell to deliver current as measured in ampere hours.

This must not be taken as an argument against the use of the "flash" test to indicate depreciation since the great simplicity and convenience of this test amply justify its use. Where it is used, however, it should be borne in mind that it indicates a more rapid falling off in capacity than is actually the case, and the injustice which may thereby be done to the cell is especially great in the smallest sizes.

It is of interest to note that in the first three curve sheets the graphs *A* and *B* are divergent at their lower ends, while for the smallest size of cell in curve 4 the two lines converge. This is



Curve No. 4.

explainable by the fact that as the flash becomes zero the capacity must also approach zero, and in curves 1, 2 and 3 this convergence would be noted if the tests were carried further.

It has been pointed out in previous discussions held before this Society that any test on a dry cell will show the condition of a dry cell only at the time it is tested, without indicating what the condition of the cell will be at some future date. While this is undoubtedly true, it is nevertheless true that shop and laboratory tests accompanying uniform methods of manufacture may furnish the means of foretelling with reasonable accuracy the future per-

formance of a dry cell. By the use of certain accelerated tests, of which the "flash" test is a type, it is possible to watch and control the uniformity of the product.

While the "4-ohm" test as used in this report cannot be considered as a standard method in view of the recommendations of the Bureau of Standards for a "2.75-ohm" test, nevertheless the curves here presented may be taken as representing fairly well the true relationship between the "flash" and the "capacity" tests. This statement is based upon a "2.75-ohm" test being made on a number of cells of various sizes and ages as a check against the "4-ohm" test.

It must also be borne in mind that either 2.75 ohms or 4 ohms per cell represents a much more rapid discharge than the small dry cells are frequently called upon to deliver in service. For example, in radio work, in which the cell shown in curve No. 3 is extensively employed, the actual current requirements are very low. A resistance of 333 ohms per cell has been adopted for testing batteries for such service. Extensive investigation has been made by our laboratories on batteries designed for this type of service, 15 cells being assembled in a battery delivering an initial voltage of 22.5 volts. In testing these batteries an end point of 17 volts is taken through a resistance of 5,000 ohms. The results derived from such tests give curves of the same general shape as shown in curve No. 3. It may be noted, however, that on very slow discharges, the decrease in capacity is shown to be less during the early part of the life of the cell than is shown where the more rapid discharge method is employed.

While it is difficult to express numerically the depreciation which might be expected from dry cells as found on the market, nevertheless an approximation may be arrived at. From the data on which this paper is based, the following table has been compiled to indicate the reasonable depreciation of the four important sizes of small cells; these being based upon the actual capacity measurements rather than upon the determination by the "flash" test.

Size of Cells		Percent Depreciation per Month	
Inches	Centimeters	First Two Months	Last Ten Months
2¼ x 1¼	5.7 x 3.2	0.0	2.1
1 13/16 x 7/8	4.6 x 2.2	0.25	4.0
1⅞ x ⅝	4.8 x 1.6	2.0	5.5
1 9/16 x 17/32	4 x 1.4	2.0	6.5

July 17, 1919.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

THE EFFECT OF AMALGAMATION UPON THE SINGLE POTENTIAL OF ALUMINUM.[1]

By Louis Kahlenberg and John A. Montgomery.[2]

Abstract.

Measuring the single potential of aluminum in a ⅓ molar solution of aluminum chloride at room temperature, by means of the calomel electrode, the writers obtained much higher values with amalgamated than with unamalgamated aluminum, due to the removal of the coat of resistant oxide by the mercury. They showed also that the measurements were actually the single potentials of the aluminum and not those of an aluminum amalgam.

The single potential of aluminum, as commonly measured in solutions of its salts, has really not been the true value, but rather that of the metal coated with a film of aluminum oxide.

Neumann[3] amalgamated aluminum by rubbing it with mercury, and then measured its potential in the three following combinations:

								Single Potential
Hg	HgCl	nKCl	||	⅓	Molar[4] $Al_2(SO_4)_3$	|	Al	1.04 volts
"	"	"	||	⅓	" $AlCl_3$	|	Al	1.01 "
"	"	"	||	⅓	" $Al(NO_3)_3$	|	Al	0.77 "

The values recorded are the total E. M. F.s of the cells less 0.56 volt, the potential of the calomel electrode.

Wilsmore,[5] in collecting data on the single potentials of the metals, gave aluminum the value of 1.276, but this was on the basis of hydrogen taken as zero and was calculated from the results of Neumann.

[1] Manuscript received June 25, 1919. Extract of a portion of a thesis submitted for the degree of Ph.D. at the University of Wisconsin.

[2] Univ. of Wisconsin, Madison, Wis.

[3] B. Neumann: Zeit. Phys. Chemie (1894), **14**, 217.

[4] One-third of the molecular weight of the salt, in grams per liter of solution.

[5] Wilsmore: Zeit. Phys. Chemie (1900), **35**, 291.

Results which are less than one-half the magnitude of those of Neumann are obtained when the potential of aluminum is measured in the salt solutions mentioned above, the aluminum not being amalgamated. This has been shown to be the case in the work which Mott[6] did, the sulphate yielded 0.4 volt, the nitrate 0.22, and the chloride 0.361. After one hundred hours the values had dropped to 0.13, —0.03 and 0.217 volt respectively. By using normal sodium hydroxide as the electrolyte Mott succeeded in obtaining much higher values, the maximum being 1.12 volts, for less than one hour, falling to 0.888 volt in five hours and rising to 1.09 volts at the end of one hundred hours. These high results are no doubt due to the solubility of aluminum oxide coating in the alkali.

TABLE I.

Analyses of the Aluminum.

Before casting:	Percent Residue	Percent Copper	Percent Iron	Percent Aluminum by Diff.
Kahlbaum's wire.....	0.263	none	0.50	99.23
Aluminum Company.	0.16	0.18	0.22	99.44
After casting:				
Kahlbaum's wire.....	0.092	none	0.52	99.38
Aluminum Company.	0.13	0.12	0.23	99.52

EXPERIMENTAL.

Two different samples of aluminum were used, but neither was pure. One sample, in the form of wire, came from Kahlbaum, while the other sample came from the Aluminum Company of America. Unless otherwise stated, all of the potential data to follow were obtained by using the aluminum from Kahlbaum in the form of wire; however, some measurements were made upon the wire after it had been melted and cast in a graphite mould. Potential measurements were made upon the aluminum from the Aluminum Company of America only after it had been recast in a graphite mould.

The analyses of the two samples of aluminum both before and after casting are given in Table I.

[6] W. R. Mott: Dissertation on "Electrolytic Deposition of Aluminum," University of Wisconsin, 1903.

All of the potential measurements made in this investigation were taken in a ⅓ molar aluminum chloride solution and at room temperature. The first measurements were made with Kahlbaum's wire, which had not been previously polished. The results obtained were quite constant when compared with the results obtained under other conditions of measurement, as will be seen later. Table II gives a few of the results for the unpolished wire, the time of immersion being indicated only at the end of the measurements for each series. The results are always expressed in volts, obtained by subtracting from the measured E. M. F. the value of the calomel half cell, +0.56 volt.

TABLE II.

Change of Potential of Unamalgamated Aluminum with Time.

Minutes	Series 1 Unpolished. Volts	Series 2 Unpolished. Volts	Series 3 Polished. Volts
..	0.242	0.245	0.32
..	0.243	0.244	....
..	0.243	0.246	....
5	0.261	0.249	0.29
6	....	0.250	....
60	....	....	0.24

It will be seen by reference to series 3 in the table below that when some means of removing the film of oxide is employed higher results are obtained. These higher potentials are not permanent, but gradually fall to that of a specimen which has not been polished. This fall of potential for the polished wire is quite indicative of the formation of a protective film.

In order to remove the oxide film more effectively amalgamation was resorted to, the specimen being amalgamated electrolytically. Neumann says that he was unable to amalgamate electrolytically; however, no difficulty was encountered in this investigation when using a saturated solution of mercuric chloride as the electrolyte. Just as soon as the cathode was thoroughly coated with mercury, it was removed from the bath, washed with distilled water, and wiped with a clean piece of filter paper. It was then hurriedly connected with the calomel half cell and the

E. M. F. measured. It was found very desirable to take the readings as soon as possible after removing the specimen from the amalgamating bath.

The data obtained from some of the first measurements made on the amalgamated aluminum wire from Kahlbaum are plotted in Fig. 1. The time is recorded from the time of immersing the sample of aluminum in the aluminum chloride solution for

TABLE III.

The Effect of Repeated Immersing, Washing and Wiping Upon the Potential of Amalgamated Aluminum.

	Time		Single Potentials
	Min.	Sec.	Volts
Newly amalgamated	0	46	1.056
	2	8	1.023
	3	7	1.013
	4	13	1.009
	5	7	1.004
	6	8	1.001
	7	16	0.994
	8	16	0.985
Washed and wiped	0	15	1.030
	1	6	1.022
	3	7	1.018
Washed and wiped	0	13	1.032
	1	8	1.031
	2	7	1.032
	7	7	1.035
Washed and wiped	0	15	1.049
	1	6	1.051
	5	16	1.071
	7	16	1.059
	9	8	1.057
	11	6	1.055
Washed and wiped	0	13	1.049
	1	8	1.043
	3	15	1.046
	6	21	1.042

measurement. It will be noted that all of the initial readings are higher than 1.01, the value obtained by Neumann in a ⅓ molar $AlCl_3$ solution. These curves are all extended to zero time and give values, in the cases of the higher readings, of about 1.09 to 1.1 volts at zero time.

The effect of repeated immersing, washing and wiping of the specimen was next tried. The wire, after being amalgamated, was immersed for measurement with the calomel half cell, and

TABLE IV.

A Continuation of the Data Given in Table III, the Aluminum Having Been Freshly Amalgamated.

	Time Min.	Time Sec.	Single Potentials Volts
Newly amalgamated.....	0	14	1.073
	1	10	1.066
	3	7	1.055
	8	9	1.055
Washed and wiped.....	0	16	1.077
	1	10	1.068
	2	25	1.069
	3	25	1.071
	4	5	1.075
	6	5	1.072
Washed and wiped.....	0	10	1.042
	2	6	1.052
	4	6	1.059
	6	5	1.067
	9	5	1.062
Washed and wiped.....	0	15	1.059
	2	5	1.063
	5	17	1.063
Washed and wiped.....	0	17	1.064
	3	14	1.072
	5	12	1.075
	12	8	1.065

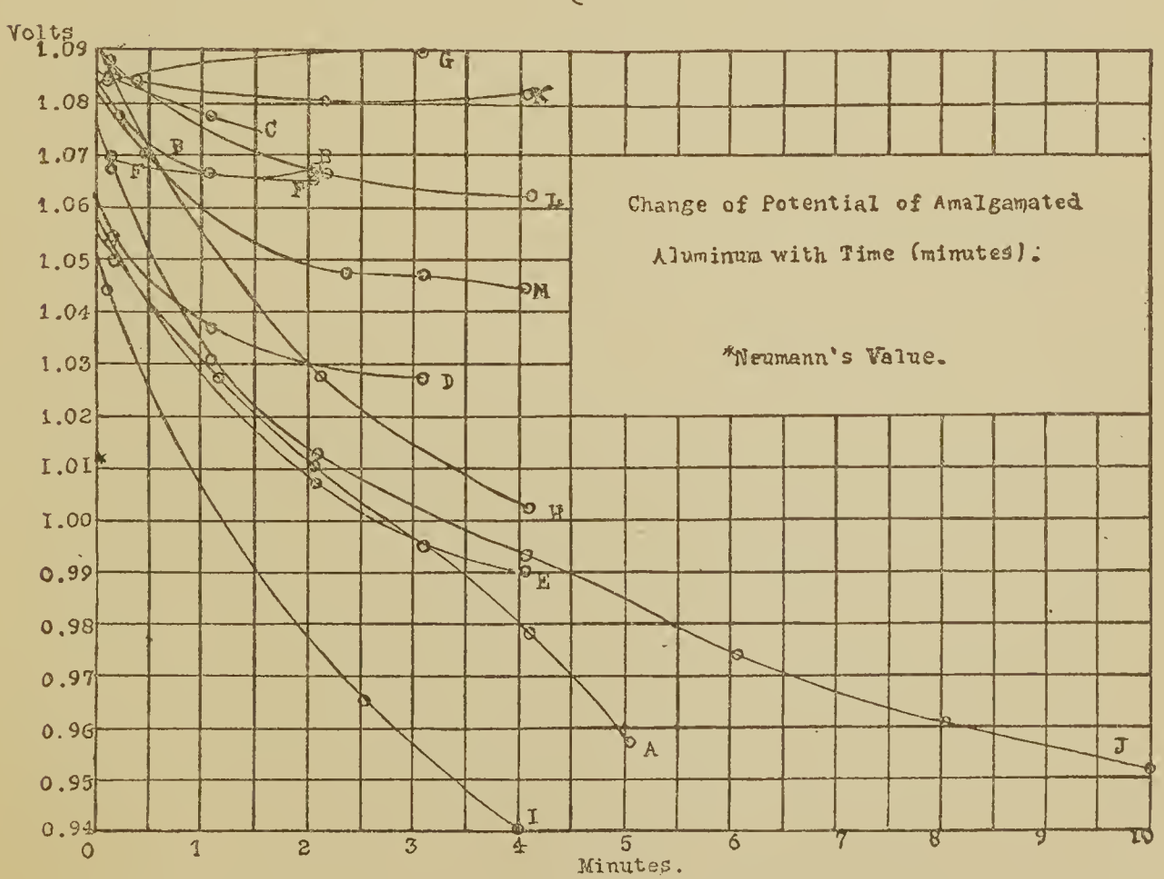


FIG. 1.

after a series of time-volt readings was taken, the wire was removed, washed with water, wiped with a clean filter paper, and then re-immersed in the aluminum chloride solution and the potential again measured. This procedure was repeated four or five times with only one amalgamation. These results are tabulated in Table III.

The electrode was next removed from the aluminum chloride and freshly amalgamated, after which it was replaced in the same portion of electrolyte. A second series of measurements obtained is given in Table IV.

TABLE V.

A Continuation of the Data Given in Table IV, the Aluminum Having Been Amalgamated a Second Time. The Time for Making the Initial Reading is Also Reduced.

	Time Min.	Time Sec.	Single Potentials Volts
Newly amalgamated.....	0	9	1.066
	1	6	1.034
	2	7	1.024
	3	11	1.022
	4	6	1.022
Washed and wiped.....	0	9	1.084
	1	6	1.068
	2	6	1.071
	3	4	1.073
	6	0	1.073
Washed and wiped.....	0	6	1.087
	1	7	1.081
	2	7	1.073
	3	6	1.071
	6	8	1.054
Washed and wiped.....	0	5	1.089
	1	27	1.053

The results obtained after the second amalgamation were somewhat higher than those obtained after the first amalgamation; however, the values fluctuate. It was thought that the potentials might be still higher after a third amalgamation, but such was not the case, for they were about the same as given in Table IV.

These experiments were repeated, using the same electrode and a new portion of electrolyte, but the results obtained did not present anything new. With the exception of the last readings in the first part of Table III, all of the readings in Tables III and IV are higher than the value found by Neumann.

The electrode was removed from the electrolyte, polished with emery paper and re-amalgamated, and then used for another series of readings, given in Table VI.

Table VII is a continuation of Table VI, the wire being again polished with emery and amalgamated.

Usually the potential of the aluminum fell with the time of immersion in the aluminum chloride solution, therefore, an attempt was made to reduce the time elapsing between the time of immersing the aluminum in the aluminum chloride solution and the taking of the initial reading. The results that were obtained with this point in view are given in Tables V, VI and VII. A new piece of Kahlbaum's wire and a new portion of

TABLE VI.

A Continuation of Table V, the Aluminum Having Been Re-amalgamated.

	Time		Single Potentials
	Min.	Sec.	Volts
Polished and Amalgamated.....	0	9	1.073
	0	36	1.053
	1	0	1.036
	1	35	1.022
Washed and wiped.....	0	7	1.081
	0	26	1.070
	0	45	1.063
	1	38	1.059
Washed and wiped.....	0	10	1.071
	0	30	1.064
	0	47	1.064
	1	30	1.064

electrolyte were used for these measurements. Forty seconds seemed to be about the minimum time in which the electrode (aluminum wire) could be removed from the amalgamating bath, washed, wiped and placed in the aluminum chloride solution.

It is evident from the foregoing data that it is very difficult to obtain constant readings. In many instances the shorter the period between immersion and the first reading, the higher the result. The highest value given in any of the above data is a five-second reading given in Table V, it being 1.089 volts. This value is very close to the values found in Fig. 1 by extrapolating the curves *B, C, G, H, K, L,* and *M* to zero time. Since these values are all of the same order of magnitude it was decided to

try to obtain instantaneous readings. This could only be accomplished by setting the potentiometer before immersing the specimen in the electrolyte for measurement. The potentiometer was set at 1.65 and then, just as soon as the electrode was immersed for measurement, the reading was made. Since the readings thus obtained were "instantaneous" or as nearly so as possible, no time is recorded in Table VIII. These readings were all made within less than five seconds after immersion. At the time of measurement the galvanometer needle would first rise to a maximum, which is the recorded value in Table VIII, and within two seconds after reaching this maximum a rapid fall of potential resulted.

TABLE VII.

A Continuation of Table VI, the Aluminum Having Been Re-amalgamated.

	Time		Single Potentials
	Min.	Sec.	Volts
Newly amalgamated	0	9	1.075
	0	32	1.062
	0	55	1.056
	1	18	1.053
Washed and wiped	0	6	1.080
	0	27	1.070
	0	43	1.071
Washed and wiped	0	4	1.084
	0	25	1.073
	0	46	1.074

Some of the Kahlbaum aluminum wire was next melted and then cast in a graphite mould. The maximum potential which could be attained, under the same conditions as those used in obtaining the data for Table VIII, was 1.06 volts. This value was obtained a number of times. A casting of the aluminum from the Aluminum Company of America, which it will be recalled contained a small amount of copper, gave a maximum potential of 1.01 volts. This value is the same as the value obtained by Neumann. It will be shown in a later paper[7] presented at the same meeting that a small amount of copper in aluminum produces a marked effect upon the potential of aluminum.

[7] The Effect of Amalgamation Upon the Single Potentials of the Binary Alloys of Aluminum with Copper, Zinc and Nickel. This volume, page 289.

The question that now arises is: What is the effect of the mercury? Are we measuring the potential of an aluminum amalgam or are we obtaining the potential of aluminum which has had its resistant oxide coating removed by the mercury?

The potentials of zinc, copper and nickel in a ⅓ molar aluminum chloride solution are practically unaltered by amalgamation, whereas the potential of aluminum is greatly increased for a short period of time. After an hour or so of immersion, however, the potential of the amalgamated aluminum is practically the same as the potential of unamalgamated aluminum. In view of the data already given, perhaps the most interesting results of all are the potentials of amalgamated and unamalgamated aluminum wire in a ⅓ molar sodium hydroxide solution. Since the potential of aluminum is greatly changed by amalgamation when it is measured in aluminum chloride solution one would expect, if we assume that we are measuring the potential of an aluminum amalgam, that the potential of amalgamated aluminum in the sodium hydroxide would be quite different from the potential of unamalgamated aluminum, but such was not the case, for we obtained a value of 1.17 volts in both cases. Mott obtained a value of 1.12 volts for unamalgamated aluminum in normal sodium hydroxide solution.

TABLE VIII.

Potentials of Amalgamated Aluminum Obtained Within Less than Five Seconds After Immersing in the Aluminum Chloride Solution.

	Single Potentials Volts
Polished and then amalgamated	1.090
Washed and wiped	1.087
" " "	1.085
" " "	1.077
Polished and then amalgamated	1.081
Washed and wiped	1.091
" " "	1.088
" " "	1.090
" " "	1.086

Almost everybody who is familiar with the chemistry of aluminum knows that amalgamated aluminum corrodes badly in the air. The authors do not recall having ever seen any photo-

graphs showing the rate of this corrosive action, and have therefore made photographs to show this action. (See Figs. 2-5.) Fig. 2 was taken two minutes after amalgamation, Fig. 3 two minutes later, Fig. 4 six minutes later, and Fig. 5 at the end of one hour.

FIG. 2. Two-minute Exposure.

FIG. 3. Four-minute Exposure.

DISCUSSION AND CONCLUSION.

Since the wire from Kahlbaum contained a greater percent of iron and insoluble material than did the sample from the Aluminum Company of America, the only logical reason to offer for the lower potential in the case of the latter sample is the other impurity, namely copper. In a later paper[8] it will be shown that this is doubtless the case. An unsuccessful attempt was made to obtain copper-free aluminum from the Aluminum Company of America.

That the effect of amalgamation is not a permanent one is shown by the time-volt curves plotted in Fig. 1. These curves also show that the effectiveness of amalgamation does not decrease at a definite speed, for the slopes of the different curves vary. In only one instance, curve *G*, is the potential value higher

[8] *Ibid.*

after several minutes than the value for zero time. By comparing the time and initial readings in Table III with the time and initial readings in Tables IV, V, VI and VII, it will be seen that in general the shorter the time between immersion of the electrode in the aluminum chloride solution and the taking of the first reading the higher the result.

There are several reasons that might be ascribed for the lower values obtained by Neumann, such as: (1) crude apparatus, (2) poor amalgamation, (3) physical state of the metal (this was not

FIG. 4. Six-minute Exposure.

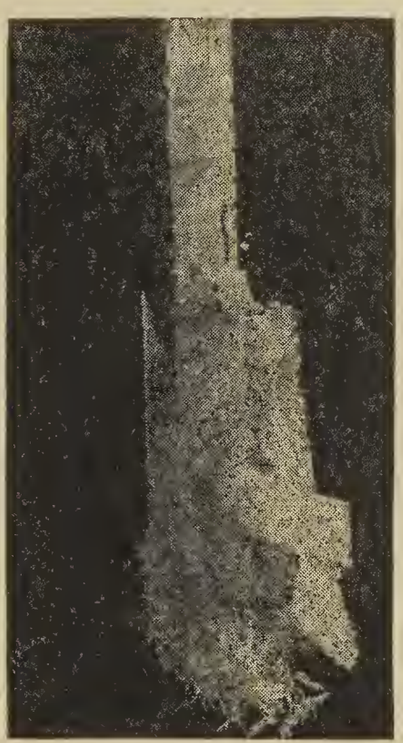


FIG. 5. Sixty-minute Exposure.

stated in his work), and (4) impurities in the metal. It has been shown that as low as 0.12 percent of copper lowers the potential of aluminum to the value obtained by Neumann.

If, by amalgamation, the potential of the amalgam rather than that of the aluminum were measured, there would be quite a difference in the potentials of amalgamated and unamalgamated aluminum in sodium hydroxide solution. Since, however, the amalgamated aluminum gives the same potential in sodium hydroxide as the unamalgamated aluminum, and since the potentials of zinc, copper, and nickel also are changed to only a very slight degree, if any, by amalgamation, and further, since the

potential of amalgamated aluminum in ⅓ molar aluminum chloride solution rapidly declines to the value of that of unamalgamated aluminum in the same electrolyte, it follows that it is not the potential of aluminum amalgam but rather that of aluminum which is measured when aluminum amalgam is placed in the electrolyte. The probable explanation for the relatively high initial potential of amalgamated aluminum is that the mercury loosens the tightly adhering film of aluminum oxide on the specimen of aluminum, thereby permitting the underlying metal to amalgamate with the coating of mercury. The potential that is then obtained is the potential of the aluminum passing from the amalgam into the aluminum chloride solution. After a short time the entire surface of the amalgam becomes coated with a thick layer of aluminum oxide, thereby causing the potential to fall to the value of that of unamalgamated aluminum. When the electrode is removed from the cell and the aluminum oxide coating is washed off, a high potential again results. This has been shown to be the case in a number of the tables given above.

Since sodium hydroxide is itself a solvent for aluminum oxide, we should expect the same potential for the amalgamated aluminum as for the unamalgamated aluminum in this solution. This was found to be the case. Moreover, the potential of aluminum is identical and comparatively constant in sodium hydroxide, whether the metal is amalgamated or unamalgamated.

University of Wisconsin,
June 20, 1919.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

THE EFFECT OF AMALGAMATION UPON THE SINGLE POTENTIALS OF THE BINARY ALLOYS OF ALUMINUM WITH COPPER, ZINC, AND NICKEL.[1]

By Louis Kahlenberg and John A. Montgomery.

I. ALUMINUM-COPPER ALLOYS.

This series of alloys has been studied by a number of investigators[2] by various methods. After the present authors had found such a great change in the single potential of aluminum,[3] in a ⅓ molar aluminum chloride solution, as a result of amalgamation, they decided to see to what extent amalgamation would change the potentials of the alloys of aluminum in the same electrolyte.

Puschin compared the results that he obtained by measuring the E. M. F. of alloys with cooling curve data and found that the two methods checked very well. He did most of his work upon such alloys as Ag-Se, Ag-Te, Cu-Te, Pb-Te, Zn-Cu, Zn-Ag, and Sn-Au. He also made measurements upon the Cu-Al series, but here he met with difficulty. He said in regard to this series that the alloys are so easily oxidized that their E. M. F.s could not be measured in acid baths, and that salt solutions were also found to be unsuitable, since the E. M. F. measured was too low and

[1] Extract of a portion of a thesis submitted for the degree of Ph.D. at the University of Wisconsin. Manuscript received July 29, 1919.

[2] C. H. Ivinson: Physical Properties, Metal Industry, **11**, 340-341.
LeChatelier: Freezing-Point Curve, Bull. de la Soc., d'Enc. (1895), 569.
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Jour. Russ. Phy. Chem. Soc., **39**, 528-556
W. Broniewski: Electrical Conductivity, Thermo-electric Power, Coefficient of Temp. and the E. M. F., Annales de Chemie et Phys., 8th Ser. (1912), **25**, 5.
Curry and Woods: Mechanical Properties, Jour. of Phy. Chem. (1907), **2**, 461.

[3] "The Effect of Amalgamation Upon the Single Potential of Aluminum." This volume, page 277.

inconsistent. He used the combination Al | $Ca(OH)_2$ | $AlCu_x$, since the single potential of aluminum in calcium hydroxide is much higher than in salt solutions.

Broniewski has made the most recent electrical measurements upon the alloys of aluminum. His work included such measurements as the electrical conductivity, thermo-electrical power, coefficient of temperature, and the E. M. F. He claims to have obtained similar results by all four methods. Fig. 1 is a graph of his E. M. F. results upon the Al-Cu series of alloys, the heavy line representing the maximum readings, while the dotted line indicates the minimum readings. The combination that he em-

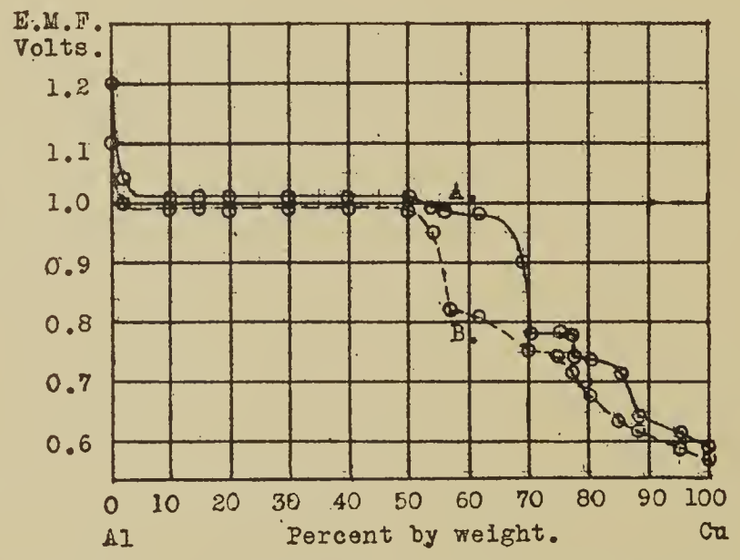

FIG. 1. E. M. F. of the Combination C | NH_4Cl, $AlCl_3$ $CuCl_2$ | Al-Cu Alloy. A = Maximum Values. B = Minimum Values. (W. Broniewski—Ann. de Chimie et de Phys., **25**, 93, 1912.)

ployed in making his measurements is as indicated in the graph.

In measuring the potentials of alloys, Laurie[4] used sodium chloride as his electrolyte and the more noble metal of his alloy as his second electrode. This method was criticized by Ostwald,[5] since he believed that as a result of the solution of the alloys the concentration of the electrolyte changed, thus causing a variation of the E. M. F. of the cell. Herschkowitsch,[6] in measuring the potentials of alloys, used as his second pole the less noble metal of the alloy and as his electrolyte a salt solution of this metal. Reinders[7] combined the views of Laurie and Herschko-

[4] M. Laurie: Chem. Soc. Jour., Trans. (1894), **65**, 103.
[5] M. Ostwald: Zeit. Phys. Chem. (1895), **16**, 749.
[6] M. Herschkowitsch: Zeit. Phys. Chem. (1899), **27**, 123.
[7] M. Reinders: Zeit. Phys. Chem. (1906), **54**, 609.

witsch, claiming in so doing that, since both metals of a binary alloy were passing into solution to a greater or less extent, a mixture of the metals composing the alloy should be used as the electrolyte. He used the less noble metal of the alloy as his second pole. Broniewski said that all of these difficulties could be eliminated by using an electrolyte not common to either of the metals in the alloy. He claimed to have eliminated any possible polarization, a point which he considered still more important. In his E. M. F. measurements, Broniewski made a cell by using a piece of graphite for the anode and the alloy as the cathode, the electrolyte consisting of a saturated solution of ammonium chloride to which had been added a two or three percent salt solution of the metals composing the alloy. Manganese dioxide was added to eliminate any possible polarization. The entire combination was in the form of a dry cell.

EXPERIMENTAL.

1. The Single Potential Measurements. The method was the same as that used in measuring the single potentials of aluminum, employing a Leeds-Northrup potentiometer, a zero galvanometer, and a calomel half cell. The electrolyte was a $\frac{1}{3}$ molar solution of aluminum chloride.

In order to show the effect of amalgamation upon the single potentials of the alloys, measurements were first made upon the unamalgamated alloys and then upon the same alloys after they had been amalgamated electrolytically.

Before making any potential measurements upon the unamalgamated alloys, they were polished with emery paper and then washed with distilled water and wiped with a clean piece of filter paper. After the completion of these measurements, the alloys were amalgamated and a second series of measurements were made. In general, the alloys rich in copper gave readings that were much more constant than those rich in aluminum. This was true whether the specimens were unamalgamated or amalgamated.

In making measurements upon the amalgamated alloys rich in aluminum, practically the same difficulties were encountered as with amalgamated aluminum. The first readings taken after

amalgamating the specimens were usually considerably higher than the succeeding readings. It was also true that the shorter the time intervening between the time of immersion in the aluminum chloride and the taking of the first reading the higher that reading. This was due, no doubt, to the rapid corrosion of the amalgamated alloys which contained more aluminum than copper. In order to obtain constant results, "instantaneous" readings were resorted to. The term "instantaneous" refers to measurements which were made within less than five seconds after placing the alloys in the aluminum chloride solution for measurement. This was only possible, of course, by making a number of preliminary measurements to ascertain the E. M. F. to be expected and then setting the potentiometer at that value. Of course, the alloy was connected to the potentiometer before immersing the former in the aluminum chloride. The specimens were always washed with distilled water, by means of a wash bottle, and wiped with clean filter paper before immersion.

Some of the measurements made on two of the typical alloys of the series containing excess of aluminum are given in Table I. The volts tabulated are the E. M. F. of the combination, Alloy | N $AlCl_3$ | Calomel electrode less +0.56 volt, the value of the calomel electrode. The time recorded in the table is the period elapsing between the time of immersion in the aluminum chloride solution and the taking of the readings. As has been stated above, the alloys containing larger amounts of copper gave results which were much more constant.

Measurements, according to this method, were made upon all of the alloys of the series. It is very evident from the data of Table I that one may obtain a great variety of results. In order to have some agreement between the measurements for the alloys of the series so that the potential curves might be plotted, the maximum potential value obtained for each alloy was used. Thus in Fig. 2 are plotted curves corresponding to these maximum readings. For those alloys containing less than 50 percent of copper, the heavy line indicates the values obtained by amalgamation and the dotted line the maximum values obtained for the unamalgamated samples. For the alloys containing more than 50 percent of copper the heavy line represents the values for the unamalgamated samples and the dotted line the values obtained for

TABLE I.

Change of Potential with Time for Two Typical Alloys Which Were Relatively Rich in Aluminum.

Percent of Copper	Time Min.	Time Sec.	Single Potentials Unamalgamated. Volts	Single Potentials Amalgamated. Volts	Remarks
4.90	*	*	0.355	....	Polished, washed and wiped after each reading.
	*	*	0.356	....	
	*	*	0.346	....	
	*	*	0.344	....	
	*	*	0.344	....	
	0	27	....	0.653	Newly amalgamated
	1	17	....	0.515	
	0	15	....	0.763	Washed and wiped
	0	51	....	0.740	
	1	15	....	0.737	
	2	4	....	0.727	
	2	30	....	0.725	
	Instantaneous		....	0.790	Re-amalgamated
	"		....	0.710	Washed and wiped
	"		....	0.710	" " "
	"		....	0.790	Re-amalgamated
22.48	0	15	0.293	....	No polishing
	0	58	0.263	....	
	2	23	0.278	....	
	3	30	0.278	....	
	0	19	0.335	....	Cleaned with emery paper
	0	43	0.316	....	
	1	5	0.307	....	
	2	6	0.291	....	
	3	5	0.286	....	
	5	0	0.286	....	
	0	17	0.330	....	Re-cleaned with emery paper
	0	49	0.334	....	
	1	27	0.319	....	
	2	14	0.316	....	
	3	24	0.314	....	
	0	39	....	0.520	Newly amalgamated
	1	3	....	0.530	
	2	10	....	0.500	
	5	8	....	0.500	
	0	14	....	0.590	Washed and wiped
	1	4	....	0.490	
	3	8	....	0.500	
	0	7	....	0.631	Washed and wiped
	0	49	....	0.512	
	2	5	....	0.514	
	6	7	....	0.501	
	21	10	....	0.518	
	30	0	....	0.518	
	Instantaneous		....	0.653	Washed and wiped
	"		....	0.657	
	"		....	0.664	
	"		....	0.661	

* The time was not recorded.

the amalgamated specimens. These data are tabulated in Table III.

It is evident from Table I that the potentials of the alloys when amalgamated or unamalgamated did not increase as a result of an accumulation of hydrogen upon the specimens, for in both cases the potentials of the alloys decreased with the time of immersion in the aluminum chloride solution. This was not true

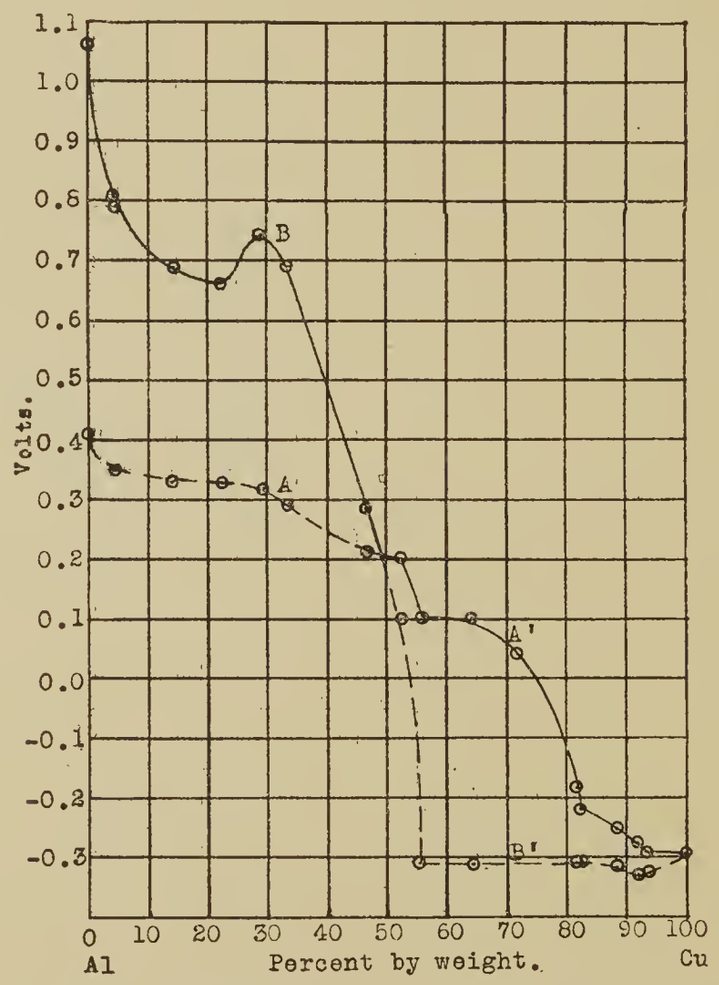

FIG. 2. Maximum Potentials of Al-Cu Alloys in *N* $AlCl_3$ Solution.

for all of the alloys of aluminum with copper, however, as will be noted by reference to Table II. In this table we see that those alloys which contained more than 50 percent of copper were polarized. The alloy containing 55.57 percent of copper did not polarize to any great extent when unamalgamated, but after it had been amalgamated the polarization was very perceptible when immersed in the aluminum chloride solution. The polarization of the alloy containing 82.25 percent of copper was greater for the unamalgamated alloy than for the alloy after it had been

amalgamated. These results are very characteristic of the measurements made upon the alloys in the different portions of the series. It will also be noted from the results given for these two alloys that by shaking the alloys while the potential measurements were being made, very constant values were obtained. It is the values obtained while the specimens were being shaken that are recorded in Table III. The alloys were tapped rapidly against the side of the container which held the aluminum chloride solution, in which the measurements were made.

TABLE II.

Change of Potential with Time for Two Typical Alloys Which Were Relatively Rich in Copper.

Percent of Copper	Time		Single Potentials Unamalgamated. Volts	Single Potentials Amalgamated. Volts	Remarks
	Min.	Sec.			
55.57	0	37	0.103	....	Polished
	6	20	0.108	....	
	6	35	0.104	....	Shaking
	10	0	0.107	....	
	10	30	0.104	....	Shaking
	25	0	0.102	....	Shaking
	1	0	....	—0.315	Shaking
	1	30	....	—0.313	Shaking
	5	0	....	—0.150	
	5	30	....	—0.309	Shaking
	6	30	....	—0.314	Shaking
	21	0	....	—0.146	
	22	0	....	—0.318	Shaking
82.24	0	20	—0.229	....	
	1	5	—0.197	....	
	1	45	—0.221	....	Shaking
	2	35	—0.194	....	
	3	0	—0.196	....	
	11	40	—0.141	....	
	12	0	—0.224	....	Shaking
	0	30	....	—0.304	
	6	30	....	—0.303	
	7	30	....	—0.306	Shaking
	8	30	....	—0.307	
	9	0	....	—0.306	Shaking

Since all of the samples were chill cast, some of them were annealed at 450° C. for an hour and then their potentials measured again, but the results were practically the same as they were before annealing.

2. Specific Gravities and Other Physical Properties. The specific gravities for the alloys of the series are given in Table III. These results were calculated from the weight of the alloy in air and its loss in weight when suspended in water. The specimens were suspended from the balance beam by means of a vaselined silk fiber. Of course, the weight of the fiber and hook was deducted. The specific gravity curve is plotted in Fig. 3.

So far as the strength of these alloys is concerned, it was found that at either end of the series the castings were tough and strong,

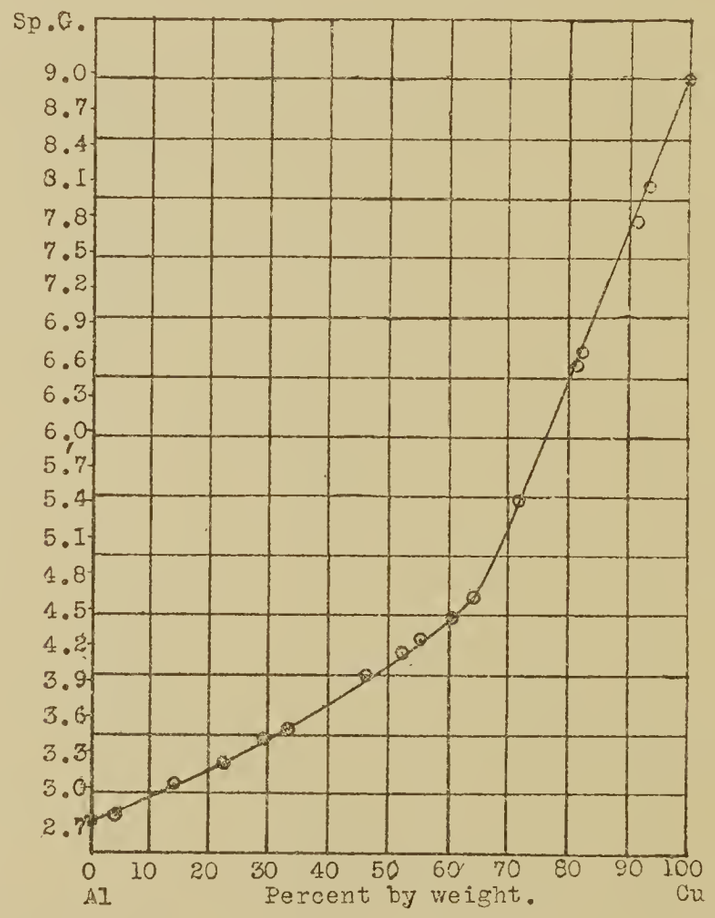

FIG. 3. Specific Gravities of Aluminum-Copper Alloys.

but in the central portion of the series they were very brittle. The 55.57 percent of copper alloy could be easily broken with the hands and could hardly be clamped in the vise sufficiently tight to permit filing. Care had to be used in polishing the alloys of the central portion of the series with emery paper so as not to break them. The 70-80 percent of copper alloys were hard and very brittle. From 80 to 100 percent of copper the brittleness rapidly decreased and the alloys became tough. Alloys of aluminum which contain more than 85 percent of copper are, unlike

those of the central portion of the series, easily polished. They are suitable for use in the arts.

All of the alloys are white up to 82 percent of copper, where they become yellow. The 88 percent of copper alloy has about the same color as gold, while those above 90 percent of copper are more red like pure copper. The alloys containing from 60 to 90 percent of copper tarnish badly in the air after exposure for two or three days.

TABLE III.

Potential and Specific Gravity Data for the Complete Series.

Weight Percent of Copper	Single Potential Unamalgamated. Volts	Single Potential Amalgamated. Volts	Specific Gravities
.0	0.41	1.06	2.70
0.15	0.41	1.01	2.70
4.20	....	0.81	2.75
4.90	0.35	0.79	2.76
14.92	0.33	0.69	3.03
22.48	0.33	0.66	3.20
29.21	0.32	0.72	3.38
33.70	0.29	0.69	3.48
46.26	0.21	0.28	3.95
52.50	0.20	0.10	4.14
55.57	0.10	—0.31	4.26
60.54	0.10	....	4.40
64.55	0.10	—0.31	4.58
71.80	0.04	....	5.41
81.50	—0.19	—0.31	6.54
82.25	—0.22	—0.31	6.65
88.31	—0.25	—0.32	7.35
91.93	—0.26	—0.33	7.75
93.30	—0.28	—0.33	8.02
100.00	—0.30	—0.30	8.94

To ascertain qualitatively the effect of increased copper content upon the rate of corrosion of these alloys, samples of numbers 4, 6, 10, 14, 17 and 19 were placed in a 5 percent sodium chloride solution. The samples were placed in separate Erlenmeyer flasks which were connected in series. Air from out-of-doors was drawn through these flasks for three weeks. Duplicate amalgamated samples were treated in like manner. Amalgamated samples 4 and 6 corroded distinctly more than the corresponding unamalgamated samples. This was shown by the fact that the

flasks containing the amalgamated specimens became much more turbid, due to the formation of aluminum hydroxide. The surfaces of the amalgamated samples also showed the effect of corrosion to a greater degree. In the cases of numbers 10, 14, 17 and 19, the results were reversed, the mercury acting as a protective coating to a certain extent, but not completely. The unamalgamated samples of 10, 14, 17 and 19 were, however, not corroded to the same extent as the corresponding samples of 4 and 6. At the end of the experiment the salt solutions were poured out and all of the samples well washed with distilled water and returned to the flasks. The stoppers of the flasks were left out and the samples thus left exposed to the air in the dry flasks. Two days later the sample of number 10, which had not been amalgamated, had completely disintegrated to a coarse powder. This sample, it will be remembered, corresponds to the alloy containing 56 percent of copper which was easily broken by the hands. Reference will be made to this again in the section on aluminum-nickel alloys.

DISCUSSION.

Usually in measuring the single potential of metals and alloys, the cell, whose E. M. F. is being measured, is allowed to come to equilibrium before making any readings; however, this method cannot be applied to aluminum and its alloys when amalgamated, for, as pointed out in another article on aluminum,[8] the potential of amalgamated aluminum rapidly falls and in a few minutes is much too low. In the case of aluminum which has not been amalgamated, the readings observed are not those of aluminum, but aluminum covered with a coating of its oxide, and they are much lower than the readings for the amalgamated aluminum. Just as this oxide coating has a considerable effect upon the potential of pure aluminum, it also likewise affects the potentials of the aluminum alloys, especially those which contain a large percent of aluminum. This point is brought out very clearly by the data in Table III, for it will be noted that those alloys containing over 50 percent of aluminum gave higher potentials when amalgamated than when unamalgamated, but samples which contained

[8] "The Effect of Amalgamation Upon the Single Potential of Aluminum." This volume, page 277.

over 50 percent of copper gave lower readings when amalgamated than when unamalgamated. As a means of obtaining the potentials of the alloys of aluminum with copper it is better to use the amalgamating method for the alloys of less than 50 percent of copper, and to make the measurements on the unamalgamated samples for those which contain more than 50 percent of copper, since as is indicated in Fig. 2 the maximum results are obtained by such a combination. The potentials obtained by this combination of methods gives the curve corresponding to the continuous

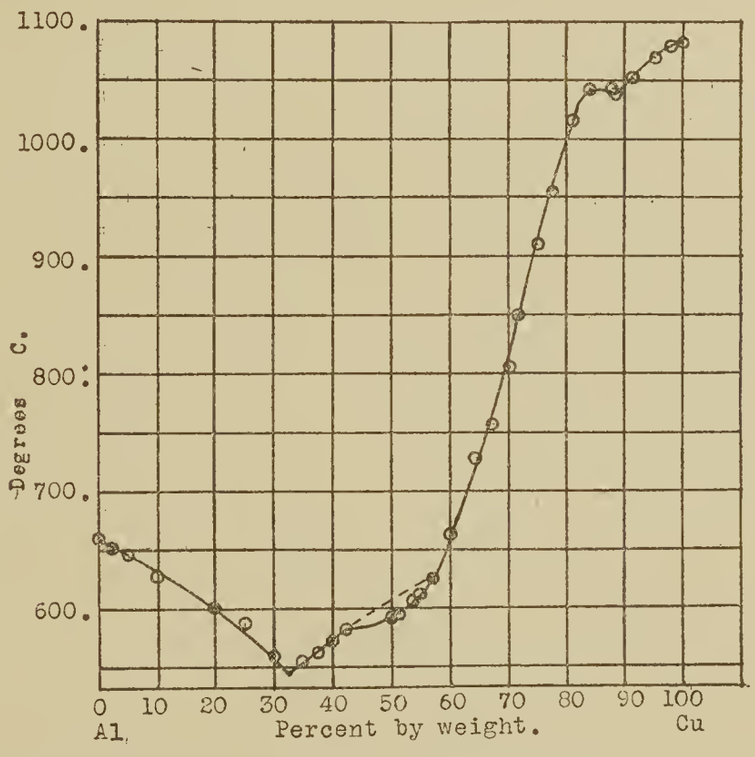

FIG. 4. Freezing-Point Curve of Aluminum-Copper Alloys. (G. C. Gwyer—Zeit. Anorg. Chemie, **57**, 117, 1908.)

heavy line in Fig. 2. The dotted line for the values below 50 percent of copper is that of the alloys which have their potentials lowered by the presence of the film of aluminum oxide. The dotted line representing the values for the amalgamated samples containing more than 50 percent of copper, is low, which may be due to the protective action of the mercury on samples which contain an excess of copper. These views are in part substantiated by the results of the corrosion tests already mentioned.

By comparing the heavy curve of Fig. 2 with the liquidus curve of Gwyer, Fig. 4, it is quite evident that the breaks in the single potential curve are much more in evidence than in the

freezing-point curve. In plotting this curve, Gwyer did not draw it in strict conformity to his data as published, but followed the course of the dotted line which makes a rather sharp break at 56 percent of copper. According to his data, the heavy curve in Fig. 4, a greater break in the curve appears at 42 percent of copper. Just why he plotted the curve to make a break appear at 56 percent of copper instead of at 42 percent of copper, where it really should fall according to his data, is not apparent from his discussion.

The breaks in the single potential curve, when the alloys below 50 percent of copper were amalgamated, as indicated by the heavy line in Fig. 2, occur at 82 percent, and 55 percent of copper, corresponding to the compounds $AlCu_2$ and Al_2Cu, if we consider these sharp changes in the curve to mean the presence of compounds. One maximum occurs in the curve, that being at 30 percent of copper which is about the same composition as the eutectic which occurs in the freezing and melting-point curves for this series of alloys. Just why this maximum should be in evidence here is not clear, unless the constituents of the eutectic have entered into a complex. In the case of the dotted curve, for the alloys containing less than 50 percent of copper, the peak at 30 percent of copper entirely disappears and the curve represents a series of solid solutions from zero to 50 percent of copper.

The value ascribed to amalgamated aluminum is 1.06 volts,[9] which is the value obtained for the wire of Kahlbaum after it had been cast in a graphite mould and then amalgamated. In preparing the alloys of aluminum the sample of aluminum from the Aluminum Company of America was used and this gave a potential value of 1.01 volts after it had been cast, but since this sample contained a small amount of copper, the value of 1.06 has been used as the correct value of cast amalgamated aluminum in a ⅓ molar aluminum chloride solution; however, this sample contained 0.52 percent of iron.

The specific gravity curve, which is plotted in Fig. 3, shows that the weights of the alloys of this series increase rather slowly from 0 percent to 60 percent of copper, and then the curve rises much more abruptly, showing a much greater increase in specific

[9] See paper by the authors, "The Effect of Amalgamation Upon the Single Potential of Aluminum," *loc. cit.*

TABLE IV.

Results and Methods of Different Investigators.

LeChatelier 1895	LeVerrier 1896	Leon Guillet 1902	W. Campbell 1904	Carpenter & Edwards 1907	Curry 1907	Gwyer 1908	Puschin 1907	W. Broniewski 1912	Present Paper
F. P. Method	F. P. Method	F. P. Method	F. P. Method	F. P. Method	M. P. Method	F. P. Method	E. M. F. Method	E. M. F. Method	Single Pot. Method
				90.6% Cu $Al Cu_4$					
87.11% Cu $AlCu_3$	90% Cu $AlCu_3$	87.11 Cu $AlCu_3$	87.11% Cu $AlCu_3$	87.11% Cu $AlCu_3$		87% Cu $AlCu_3$		87% Cu $AlCu_3$	90% Cu $Al Cu_3$
						70% Cu ? Al Cu	70% Cu Al Cu	79% Cu $Al_2 Cu_3$	82% Cu $Al Cu_2$
54% Cu $Al_2 Cu$	50% Cu $Al_2 Cu$	54% Cu $Al_2 Cu$	54% Cu $Al_2 Cu$ 48% Cu $Al_5 Cu_2$	54% Cu $Al_2 Cu$	53.7% Cu $Al_2 Cu$	54% Cu ? $Al_2 Cu$ 42% Cu $Al_3 Cu$		70% Cu Al Cu 54% Cu $Al_2 Cu$	55% Cu $Al_2 Cu$
				MAXIMA					
					87.5% Cu				30% Cu
				MINIMA					
90.5% Cu 54.5% Cu 33% Cu	92.5% Cu 62.5% Cu 33% Cu				90.5% Cu 32% Cu	88% Cu 33% Cu			22% Cu

gravity as the percent of copper increases from 60 percent to 100 percent. The alloys from 50 percent to 75 percent of copper expand on cooling and are so brittle that it is rather difficult to remove them from the mould without breaking them. The alloys in the central portion of the series are worthless from a commercial standpoint.

CONCLUSION.

In conclusion a tabulated outline, Table IV, of the results obtained by the different men who have investigated this series is given, as well as the method of study. From this table it is seen that the freezing and melting-point methods give results very much alike. The E. M. F. results and single potential measurements as obtained by three different investigators by as many different methods do not agree. Puschin located only one compound corresponding to AlCu at 70 percent of copper, whereas Broniewski obtained four compounds, $AlCu_3$, Al_2Cu_3, AlCu and Al_2Cu. In the present investigation three compounds corresponding to Al_2Cu, $AlCu_2$, and $AlCu_3$ were obtained from the curve as plotted for the measurements made upon the unamalgamated alloys, while by amalgamation two compounds Al_2Cu and $AlCu_3$ were obtained. The last two compounds are the same as have previously been obtained from thermal data by most of the investigators. In other words, by amalgamating and then measuring the single potentials of the alloys, compounds similar to those found by thermal data have been obtained, whereas without amalgamation such is not the case.

That the alloys of the series do suffer marked changes in properties at 55 percent and above 80 percent of copper is, however, quite evident. The compounds corresponding to 70 percent and 54 percent of copper that Gwyer claims to have located are marked with a question mark, since his curve, if drawn according to his data, does not show any breaks at these points.

Of all of those who have made thermal measurements upon these alloys, Gwyer is the only one who has reported a compound at 70 percent of copper.

Just as aluminum, when amalgamated, gives a much higher potential than when it is unamalgamated, so the alloys of Al-Cu containing less than 50 percent of copper have their potential

greatly increased by amalgamation. The greater the percent of aluminum, above 50 percent of aluminum, the higher the resulting potential of the alloy.

The results that have been obtained in this work by amalgamating the alloys before measuring their single potentials are quite different from those obtained from unamalgamated alloys.

II. ALUMINUM-ZINC ALLOYS.

This series of alloys is far less complicated than the Al-Cu series, for almost all of the investigators[10] are agreed that the series consists of a series of two solid solutions and a eutectic.

Broniewski[11] examined this series by the same methods as he employed in studying the aluminum-copper series which have

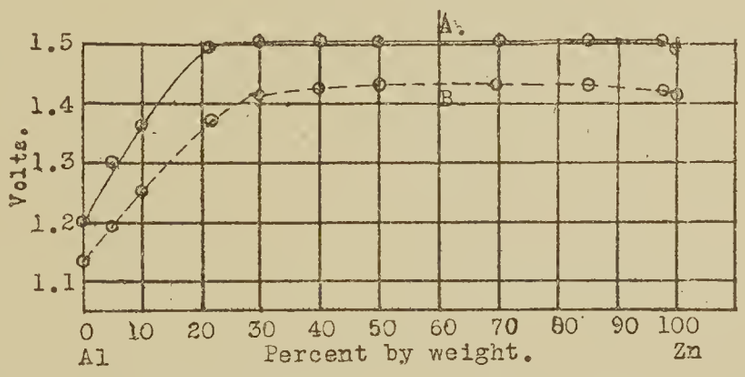

FIG. 5. E. M. F. of the Combination C | NH_4Cl, $AlCl_3$, $ZnCl_2$ | Al-Zn Alloy. A = Maximum Values. B = Minimum Values. (W. Broniewski—Ann. de Chimie et de Phys., **25**, 57, 1912.)

already been given in the previous section. Fig. 5 is a graph of his results for the E. M. F. measurements. The dotted line indicates the minimum readings and the heavy line the maximum readings. These curves do not show any evidence of a compound occurring at any point in the series. The curves do show two particular features, however, one is that a number of the alloys in the series gave higher E. M. F. readings than either of the

[10] Heycock and Neville: Melting-Point Method, Jour. Chem. Soc. (1897), **71**, 383.
W. Campbell: Freezing-Point Method, Jour. Franklin Inst., July, Aug. and Sept., 1902.
Hector Pecheux: Solubility, Compt. Rend. (1904), **138**, 1103.
E. S. Shepherd: Physical Properties, Jour. Phys. Chem. (1905), **9**, 504.
W. Rosenhain and S. L. Archbutt: Thermal Data, Phil. Trans. Roy. Soc. of London, A **211**, 315-43; Engineering, **92**, 438-42.
Rosenhain and Archbutt: Mechanical Properties, Inst. of Mech. Eng., Parts 1 and 2, 319 (1912).
J. W. Richards: Physical Properties, Iron Age, Aug. 13, 1903.
W. F. Durnad: Physical Properties, Science, **5**, 396.
Bauer and Vogel: Physical Properties, Jour. Soc. Chem. Industry (1916), **35**, 543.
D. Ewen and T. Turner: Physical Properties, Revue Metal (1911), **81**, 730.

[11] W. Broniewski: E. M. F., etc., Ann. de Chimie et Phys. (1912), **25**, 57.

pure metals, and the second is that the E. M. F.s of the alloys rich in zinc are all higher than those rich in aluminum. The curves are not plotted from single potential measurements but are the E. M. F. results.

EXPERIMENTAL.

The aluminum used was the same as that used in the preparation of the aluminum-copper alloys. It contained 0.15 percent copper, 0.22 percent iron and 0.13 percent of insoluble silicates. The zinc used was pure, hence any detrimental influences were eliminated.

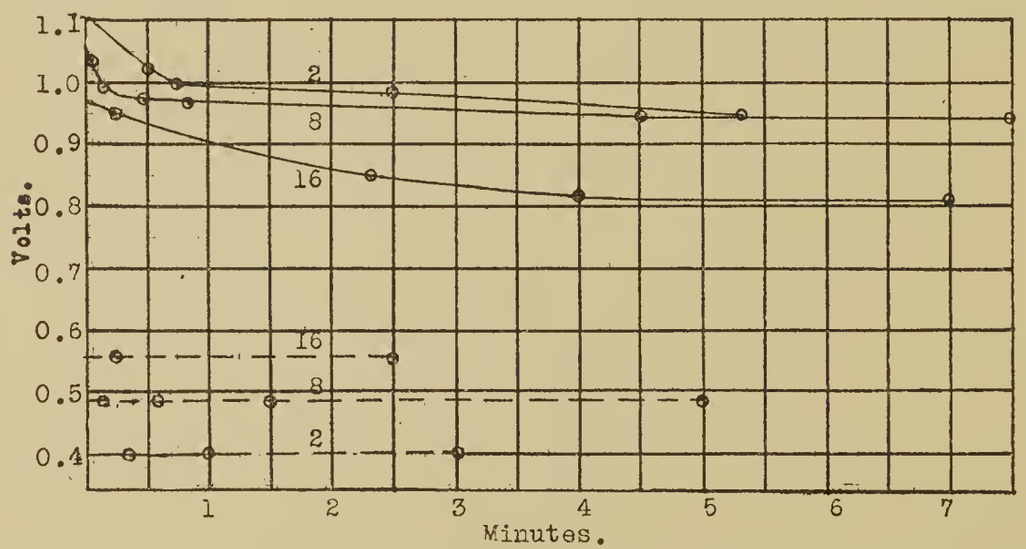

FIG. 6. Change of Potentials of Al-Zn Alloys Numbers 2, 8 and 16, with the Time. Solid Lines are the Potentials of the Alloys when Amalgamated. Dotted Lines are the Potentials of the Alloys when Unamalgamated.

1. Single Potential Measurements. The methods used in obtaining the maximum potential values for this series were just the same as those described in the section on Al-Cu. The measurements were made against a normal calomel electrode, using a ⅓ molar $AlCl_3$ solution as the electrolyte. The potentials were measured both before and after amalgamating the alloys, the amalgamation being produced electrolytically.

In order to show the change of potential with the time, in the case of both amalgamated and unamalgamated alloys, some of the readings obtained for three typical specimens have been tabulated in Table V, the corresponding graphs being given in Fig. 6. The number of each curve corresponds to the number of the

alloy. The continuous lines are the curves for the readings obtained with the amalgamated alloys and the dotted lines are the curves for the readings of the unamalgamated alloys.

TABLE V.

The Effect of Time upon the Potentials of the Amalgamated and Unamalgamated Alloys.

No. of Alloy	Time		Single Potential Amalgamated Volts	Single Potential Unamalgamated Volts
	Min.	Sec.		
2	0	22	....	0.44
	1	0	....	0.44
	3	0	....	0.44
	0	4	1.03	....
	0	9	0.99	....
	0	28	0.968	....
	0	52	0.961	....
	1	45	0.96	....
	4	30	0.943	....
	7	30	0.94	....
	104 hours*		0.53	....
8	0	8	....	0.488
	0	35	....	0.485
	1	30	....	0.483
	5	0	....	0.482
	0	17	1.024	....
	0	45	1.008	....
	2	30	0.983	....
	5	20	0.944	....
16	0	15	....	0.553
	2	30	....	0.553
	0	15	0.95	....
	2	20	0.85	....
	4	0	0.82	....
	7	0	0.81	....

* This result is taken from Table VI for the same alloy.

A few of the alloys were amalgamated and their potentials measured at intervals during a hundred-hour period. The results are tabulated in Table VI. The final potential readings obtained at the end of one hundred hours for all of the alloys thus measured are roughly those of amalgamated zinc.

The maximum values obtained for both the amalgamated and unamalgamated alloys of the entire series, as well as the differences between the two values, are given in Table VII.

Table VI.

Change of the Potentials of Several of the Amalgamated Alloys During a One-Hundred-Hour Period.

No.									
2	Pot.-Volts	1.029	0.830	0.690	0.580	0.550	0.560	0.530	***
	Time-Hrs.	**	1	6	25	54	74	104	....
6	Pot.-Volts	1.026	***	***	0.605	0.554	0.534	0.525	***
	Time-Hrs.	**	....	....	29	53	78	100	....
10	Pot.-Volts	1.031	0.722	***	0.620	0.595	0.574	0.560	0.556
	Time-Hrs.	**	2	....	20	44	64	88	113
13	Pot.-Volts	1.030	***	***	0.601	0.560	0.548	0.543	***
	Time-Hrs.	**	....	....	24	48	72	97	....
16	Pot.-Volts	0.952	0.710	***	0.530	0.528	0.552	0.521	***
	Time-Hrs.	**	2	....	21	45	72	101	....
19	Pot.-Volts	0.795	***	***	0.559	0.549	0.544	0.541	0.533
	Time-Hrs.	**	....	....	23	51	75	94	114

** Less than one hour. See Table VII.
*** No measurements were made.

2. Specific Gravities and Other Physical Properties. The specific gravities of the alloys were measured in the same manner in which those of the Al-Cu series were determined. The alloy was first weighed in air and then again when suspended under water. The specimens were suspended under the water by means of a silk fiber, the fiber having been previously greased with vaseline to prevent its increasing in weight when placed in the water. The specific gravities calculated from these data are tabulated in Table VII, with the corresponding specific gravity curve given in Fig. 7.

All of the alloys cast well. Those of high aluminum content were very ductile and could be bent almost double before breaking, but as the percentage of zinc increased the ductility decreased, and above 40 percent of zinc the alloys were quite brittle.

Samples from the alloys numbers 4, 9, 12, 15, 18 and 20 (which was pure zinc) were subject to the same corrosion test as was used on the Al-Cu series. Duplicate samples were used, one of which was amalgamated before being placed in the salt solution.

The method of carrying out this test is given in the section on Al-Cu under the heading "Physical Properties," and will therefore not be repeated here. The results of this test were that all of the amalgamated samples corroded badly and the solutions became quite cloudy with the exception of the pure zinc, and this was affected very slightly, if at all. In fact, the solution in which the amalgamated zinc had been placed gave only a slight test for zinc with potassium ferrocyanide, using uranium nitrate as an indicator. The unamalgamated samples corroded to a small degree as compared with those samples which had been amalga-

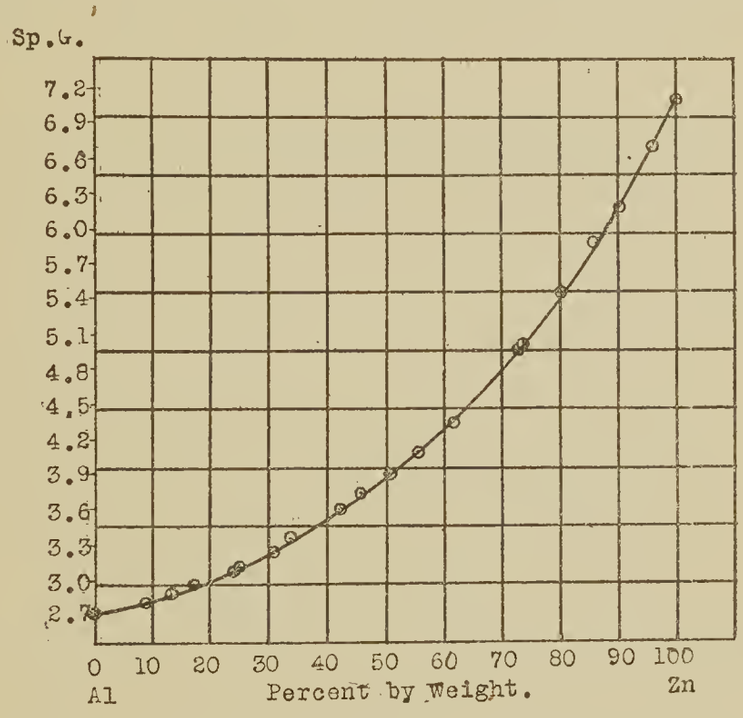

FIG. 7. Specific Gravities of Aluminum-Zinc Alloys.

mated. The solutions in which the unamalgamated alloys had been placed were not cloudy at the end of the experiment, but upon being tested showed the presence of both aluminum and zinc. There was one exception to this, however, and that was the solution containing the unamalgamated sample number 12 corresponding to 55.08 percent of zinc. This sample was badly corroded and had broken into three different pieces. This will be referred to in connection with a similar experiment for the Al-Ni series. The unamalgamated pure zinc samples gave results just like the amalgamated zinc, thus showing that amalgamation had not altered the corrosive action of the zinc.

DISCUSSION.

The heavy lines of Fig. 6 do not indicate the difficulties encountered in obtaining the maximum values for the amalgamated series. The unamalgamated specimens were comparatively easy to measure after the surface had been polished with emery cloth, washed, and wiped with a clean filter paper. In measuring the potentials of the amalgamated alloys it is very essential that the surface be well amalgamated and that the readings be taken in as short a time as possible after immersing the specimen in the aluminum chloride solution. There was only one specimen measured in the amalgamated condition whose value remained con-

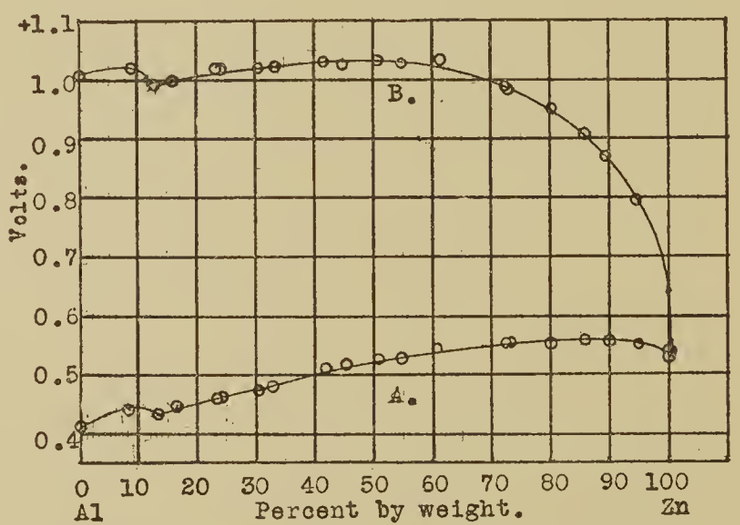

FIG. 8. Maximum Single Potentials of Al-Zn Alloys in *N* $AlCl_3$ Solution. A = Potentials of the Unamalgamated Alloys. B = Potentials of the Amalgamated Alloys.

stant for any length of time, and that was the pure zinc. It is evident from Fig. 6 that the potentials of the unamalgamated alloys changed very little during the first few minutes, while the greatest rapidity of change for the amalgamated alloys was just after immersion in the aluminum chloride solution. Table VI shows that the values of the amalgamated alloys do not become constant after a few minutes but that the values constantly decline even for a period of 100 hours, after which time the values are practically those of pure zinc, this zinc having a higher potential than aluminum coated with aluminum oxide. For this reason, if for no other, one is led to the conclusion that the correct potential values are obtained more nearly if the measurements are made as soon after immersion in the aluminum chloride solution as pos-

sible, rather than waiting for an equilibrium to be established between the alloy and the electrolyte.

The curves, plotted from the single potential results that are tabulated in Table VII, are given in Fig. 8. Curve *A* of this figure is plotted from the data obtained for the unamalgamated alloys, while Curve *B* is that of the amalgamated alloys. A slight break occurs in both curves at 13 percent of zinc, but this break stands out more prominently in Curve *B*. Since microscopic examination fails to reveal any peculiar formation at this percent of zinc other than that of a solid solution, and since the irregularity is comparatively small, we might conclude that the break in the curve is probably due to the surface condition of the alloy.

The potentials of the amalgamated series are higher than those of the unamalgamated series; however, in the case of pure zinc the difference is very small. The potential of the amalgamated zinc is only 0.01 volt higher than that of the unamalgamated zinc. The smallest difference noted between any of the alloys was 0.245 volt in the case of the 95.3 percent of zinc alloy. As the percentage of aluminum increased, the differences of potentials for the amalgamated and unamalgamated specimens constantly increased, amounting to 0.60 volt in the case of zero percent of zinc. The aluminum used contained a small amount of copper, iron and insoluble matter. According to Curve *A* of Fig. 8 the potential of pure zinc is higher than that of aluminum, if we assume that we are actually measuring the potential of aluminum rather than the aluminum coated with oxide. When amalgamated, the order of the potentials is just reversed, the value of the pure zinc remaining practically constant, while the potential of the aluminum rises from 0.41 volt to 1.01 volts. The low values in the case of unamalgamated aluminum and the alloys must be due to the film of oxide coating, for when the effect of this film is eliminated to a large extent by means of amalgamation the readings are much higher. The effectiveness of the film seems to decrease with each increased percentage of zinc. Neumann[12] gives 0.503 volt as the potential of zinc in zinc chloride, which is a little lower than we found it to be in a ⅓ molar $AlCl_3$ solution.

[12] B. Neumann: Zeit. Phys. Chemie (1894), 14, 217.

TABLE VII.

Single Potential and Specific Gravity Result in the Entire Series.

No. of Alloy	Single Potential of Unamalgamated Alloy. Volts	Single Potential of Amalgamated Alloy. Volts	Difference Volts	Specific Gravities
1	0.410	1.010	0.60	2.7
2	0.440	1.029	0.589	2.79
3	0.437	0.990	0.567	2.858
4	0.446	1.000	0.566	2.95
5	0.461	1.020	0.561	3.057
6	0.460	1.026	0.566	3.10
7	0.475	1.026	0.551	3.20
8	0.480	1.024	0.544	3.34
9	0.510	1.030	0.52	3.60
10	0.514	1.031	0.517	3.73
11	0.523	1.032	0.509	3.90
12	0.530	1.036	0.506	4.05
13	0.548	1.031	0.483	4.30
14	0.551	0.991	0.44	4.97
15	0.551	0.991	0.44	5.03
16	0.553	0.952	0.399	5.45
17	0.564	0.914	0.350	5.90
18	0.560	0.875	0.315	6.19
19	0.550	0.795	0.245	6.76
20	0.530	0.540	0.04	7.09

The question that now arises is—are we justified in assuming that by amalgamation we are able to obtain potential results which are nearer to what the potential of aluminum and its alloys would be if it were not for the coating of aluminum oxide, or on the other hand, are we measuring the voltages produced as a result of an electric couple between the aluminum and mercury, or the alloy and mercury, as the case may be? We believe that the potential results obtained on the Al-Zn series are sufficient to justify us in our assumption. Let us suppose that in the case of amalgamated aluminum we have an electrical cell of

$$Hg \mid HgCl\ N\ KCl \parallel N\ AlCl_3 \mid Al$$

with a voltage of 1.01. This value is the single potential of the amalgamated aluminum as used in this series of alloys. It contained a little copper, iron and insoluble material. As a second cell let us use the combination

$$Hg \mid HgCl\ N\ KCl \parallel N\ AlCl_3 \mid Zn.$$

We will take as the voltage of this cell the value 0.54, which is the potential of amalgamated zinc in a ⅓ molar $AlCl_3$ solution. These results, interpreted in terms of the relative positions of aluminum and zinc in the electromotive series, simply mean that aluminum with reference to mercury is more electropositive than zinc, hence aluminum is above zinc in the electromotive series. If we take as the single potentials of aluminum and zinc, the values obtained when the metals are unamalgamated, the zinc will be above aluminum, since the single potential of unamalgamated zinc is 0.53 volt while unamalgamated aluminum has a potential of 0.41 volt. These values are taken from the measurements made in ⅓ molar $AlCl_3$ solution. It is apparent that aluminum

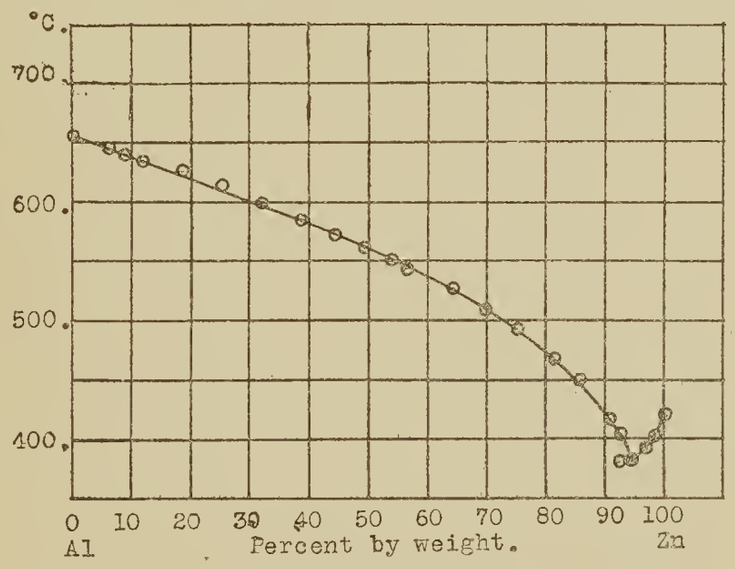

FIG. 9. Freezing-Point Curve of Aluminum-Zinc Alloys. (Heycock and Neville—Jour. Chem. Soc., 71, 383, 1897.)

cannot occupy both positions at the same time. Moreover, if the higher potential of amalgamated aluminum is due to an electrical couple, then we should reasonably expect the potential value of amalgamated zinc to be much above that of the unamalgamated zinc, but as a matter of fact the two values for zinc are practically the same. Because of these facts we believe that we are perfectly justified in using the amalgamated aluminum alloys for measuring their single potentials when the potentials of the amalgamated alloys are greater than the unamalgamated alloys.

The measurements that were made upon this series of alloys did not show any increase of potential with the time either in the case of the amalgamated or unamalgamated specimens; therefore, polarization did not have to be dealt with.

CONCLUSION.

From the preceding data we are led to the conclusion, as has already been found by other investigators, that Al-Zn alloys do not form any definite compounds, but a series of solid solutions. The eutectic located by freezing and melting-point data does not have any effect upon the potential curve.

If one wishes to obtain the potential values for Al-Zn alloys rather than those of aluminum oxide and zinc, it seems necessary that one employ some method of eliminating the oxide effect, amalgamation having been resorted to in this work.

Because of the effect of time upon the potentials of these alloys, it is very important that the measurements be made as quickly as possible after amalgamation. It has been definitely pointed out that after an immersion of a number of hours the potentials of these alloys decline to the value of zinc, the greatest change taking place just after amalgamation.

By amalgamating these alloys one obtains potentials which are much higher than the potentials of the unamalgamated alloys. When the alloys are amalgamated, the resulting potentials are all greater than that of zinc, whereas measurements made upon the unamalgamated alloys are, with few exceptions, less than the potential of zinc.

III. ALUMINUM-NICKEL ALLOYS.

Gwyer,[13] by means of thermal data, was the first to give the real constitution of the series, he having located the compounds Al_3Ni, Al_2Ni and AlNi. A plot of his results is given in Fig. 10.

Other investigators[14] have examined this series of alloys to a greater or less extent.

Broniewski[15] obtained E. M. F. data for the series which are given in Fig. 11. The method used in preparing the alloys is not given, nor does he make any mention as to why he did not obtain

[13] G. C. Gwyer: Zeit. Anorg. Chem. (1908), **57**, 133.

[14] Tissier: Physical Properties, Recherche de Aluminum, 1858.
E. S. Sperry: Physical Properties, Trans. Amer. Inst. of Min. Eng., **29**, 280.
O. Brunck: Study of Compounds in the Series, Ber. Chem. Gesell. (2) (1910), **34**, 2733.
L. Guillet: Study of Compounds in the Series, Bull. Soc. Encour. (1902), 259. Les Alliages Metalliques (1906), 942.
Campbell and Mathews: Physical Properties, Jour. Amer. Chem. Soc. (1902), 253.
Read and Greaves: Physical Properties, Jour. of Inst. of Metals (1915), **13**, 100.

[15] W. Broniewski: Ann. de Chimie et Phys. (1912), **25**, 106.

data for the central portion of the series. The probabilities are that he failed to make alloys of sufficiently desirable physical qualities to justify any attempts at measurement in the central portion of the series. He says that he failed to find any compounds in the series with the single exception of Al_3Ni.

Richards[16] gives two different methods for the preparation of the alloys of the Al-Ni series. In the first case an alloy of equal portions of aluminum and nickel was prepared and this was added to molten aluminum. The chief difficulty with this method

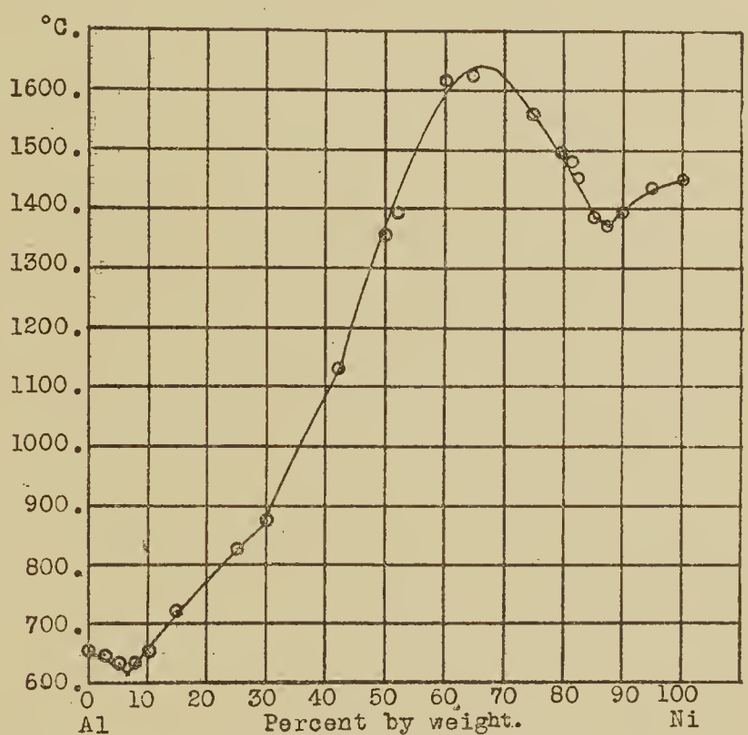

FIG. 10. Freezing-Point Curve of Aluminum-Nickel Alloys. (G. C. Gwyer—Zeit. Anorg. Chemie, **57**, 136, 1908.)

is the preparation of the fifty percent alloy. His other method consists of adding the oxide of nickel to a bath in which alumina is being reduced.

EXPERIMENTAL.

The aluminum which was used in preparing these alloys was the same as that which had previously been used in preparing the copper and zinc alloys. The nickel contained 0.43 percent of iron but no cobalt.

In the preparation of these alloys, the only type of electric furnace accessible and at the same time suitable for temperatures

[16] J. W. Richards: Iron Age, Aug. 13, 1903.

up to 1,700° C. was a carbon resistance furnace. Since clay crucibles are unsuitable for such high temperatures, another type of container had to be used. A graphite crucible could not be used alone since molten nickel will take up carbon just as iron does; therefore, electrically fused magnesia was employed as a lining for the graphite crucibles.

In producing alloys it is very essential that the metals be well mixed, otherwise different portions of the same casting will vary in properties. In producing alloys in the type of electric furnace used in this work, it would be rather difficult to stir the metals after they had melted, since the crucibles were entirely covered with granulated carbon. In order to overcome to some extent the difficulty in obtaining a good mix without stirring, advantage was taken of the difference in physical properties of the two metals being used. Pure aluminum melts at 655° C., while pure nickel melts at 1,450° C. The specific gravity of aluminum is 2.7 and the corresponding value of nickel is 8.9. The form that was used in making the first crucibles employed consisted of two pieces. The lower portion of the form was one-half inch in diameter and two and one-half inches long, while the upper portion was one and one-half inches in diameter and one inch long. By using such a combination, magnesia linings were made which had a long narrow bore at the bottom with a much larger bore at the top, thus forming a shelf on which the solid metals could be placed. The metals were so arranged on this shelf that the aluminum, which, of course, would melt first, would run into the lower portion of the crucible and then, when the nickel melted, it would run in on top of the aluminum, and since its specific gravity is much greater than that of aluminum, it would diffuse down through the aluminum and thus mix to some extent. It was thought that by breaking up the alloy thus formed and then remelting and recasting, a very good specimen could be obtained. This seems as though it would be a very good method for producing these alloys, but in practice it does not work at all. Instead of the aluminum running into the lower portion of the crucible, it oxidized so badly at the upper end of the smaller chamber that not even the nickel could run through it. Oxidization was so great that even the different globules of molten nickel were prevented from uniting with each other. The resulting con-

glomeration did not resemble an alloy in the least. A number of unsuccessful attempts were made to use this form of crucible.

During the course of experimentation it was found that whenever a piece of nickel succeeded in becoming well surrounded by molten aluminum before oxidation had proceeded too far, the nickel thus surrounded would alloy with the aluminum in immediate contact with it. The surface of the aluminum, however, was always badly oxidized. Finally the shelf type of crucible was entirely dispensed with and the entire bore of the magnesia crucible made one and one-half inches in diameter and four inches deep. In using this type of crucible the two solid metals

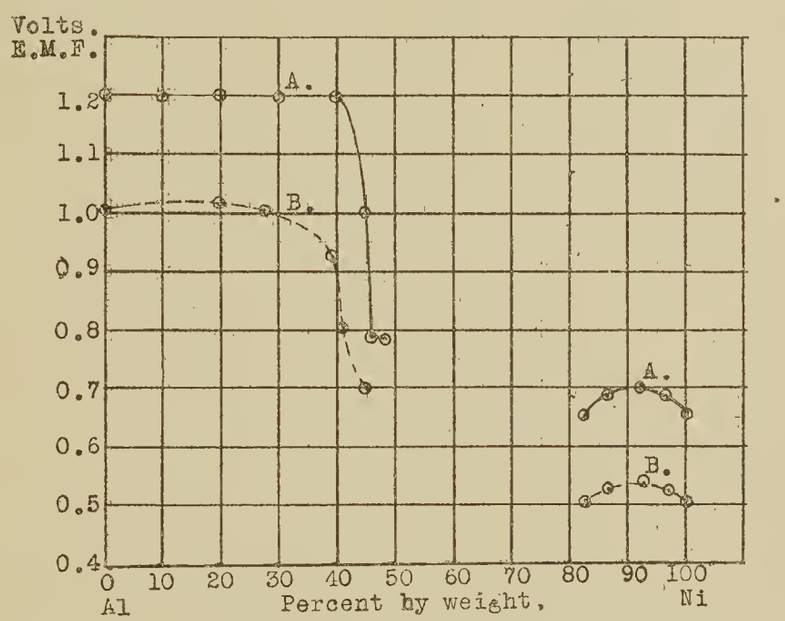

FIG. 11. E. M. F. of the Combination C | NH_4Cl, $AlCl_3$, $NiCl_2$ | Al-Ni Alloy. A = Maximum Values. B = Minimum Values. (W. Broniewski—Ann. de Chimie et de Phys., **25**, 110, 1912.)

were added in the desired proportions and heated. Such a scheme resulted in more or less alloying, but on the whole the product was quite unsatisfactory since much of the nickel was kept away from the molten aluminum by the oxide of aluminum. This method was then modified, for instead of putting solid aluminum into the crucible with the nickel, the aluminum was melted in a gas furnace and then the molten aluminum and solid nickel were put into the magnesia crucible in alternate layers. The crucible was thus filled with as many as six or seven alternate layers of aluminum and nickel, all of the nickel being surrounded by aluminum. This eliminated to a large extent the formation of a layer of aluminum oxide around the different pieces of nickel and

consequently the nickel alloyed with the aluminum. Alloys produced by this scheme were far from homogeneous; however, it was by this method that an alloy of about 40 percent of nickel was produced. Since the melting-point of this alloy was below 1,100° C. it was remelted in the gas furnace by means of two blast lamps and various amounts of aluminum were added. After the metals had liquefied they were well stirred and the alloys then cast in a graphite mould. Alloys varying from zero to 35 percent of nickel were produced in this manner.

By this same method of protecting the nickel from aluminum oxide, an alloy richer in nickel was produced; however, it was not homogeneous and would have been unsuitable for single potential measurements. Two weeks after this alloy had been made it appeared to be just as it was when first taken from the crucible, but a gentle touch with the finger showed that this was not the case, for it crumbled to pieces just as a pile of granulated cane sugar would fall to pieces if disturbed. The resulting granules of the alloy were about the same size as those of cane sugar. An analysis of this alloy showed that it consisted of 56 percent of nickel and 43.54 percent of aluminum. When first made it was hard and brittle and required a blow with the hammer to break it. Its color, like all of the alloys of aluminum with nickel, was white. According to Gwyer, the melting-point of the 56 percent nickel alloy of aluminum is a little above 1,500° C.; hence this alloy could not be remelted in the gas furnace. In the preparation of this alloy sufficient nickel was put into the crucible to make a 75 percent nickel alloy, but in the course of heating some of the nickel became surrounded with sufficient oxide to prevent the nickel from alloying with the main portion of the alloy.

1. Single Potential Measurements. The potentials of these alloys were measured both in the unamalgamated and amalgamated conditions. The measurements were all made in a ⅓ molar aluminum chloride solution against a calomel half cell. Just as has already been explained in the section on the alloys of aluminum with copper, each alloy was polished with emery paper, then well washed with water and wiped with a clean piece of filter paper before any measurements were made. After each amalgamation the alloy was carefully washed and wiped before placing it in the aluminum chloride solution. Some of the meas-

urements taken upon alloy number six, which contained 18.21 percent of nickel, are typical of the measurements made upon the other alloys of the series. These results are given in Table VIII.

TABLE VIII.

The Effect of Time Upon the Potential of a Typical Aluminum-Nickel Alloy.

Number of Alloy	Time		Single Potential Amalgamated Alloy Volts	Single Potential Unamalgamated Alloy Volts	Remarks
	Min.	Sec.			
6 18.21% Ni	0	55	....	0.233	Polished
	1	30	....	0.235	
	2	15	....	0.238	
	24	0	....	0.216	
	27	0	....	0.211	
	0	10	0.845	....	Polished
	0	30	0.857	....	
	1	0	0.857	....	
	1	30	0.840	....	
	2	10	0.806	....	
	3	0	0.766	....	
	10	30	0.278	....	
	0	15	0.840	....	Repolished
	0	40	0.860	....	
	0	55	0.852	....	
	1	10	0.840	....	

Table IX contains the maximum readings obtained for each of the alloys, both amalgamated and unamalgamated. The curves .which are plotted from these data are given in Fig. 12. The heavy line represents the maximum readings taken for the amalgamated alloys, while the dotted line represents the maximum readings taken for the unamalgamated alloys.

It has been pointed out in the section on aluminum-copper alloys that for those alloys of aluminum with copper which contained less than 50 percent of copper, the potential was not changed appreciably by shaking the alloys while the potential measurements were being made; however, with the alloys containing more than 50 percent of copper the results were different when the alloys were shaken. The measurements which were made upon the alloys of aluminum and nickel which contained less than 35 percent of nickel were very much like the results obtained for

the corresponding alloys of aluminum with copper. It was found unnecessary to shake the alloys of aluminum with nickel at the time of making the potential measurements. Pure nickel, however, polarized much more than did the pure copper, and it was necessary that the nickel be shaken during the time of measuring its potential. The same thing was true of the nickel after it had been amalgamated.

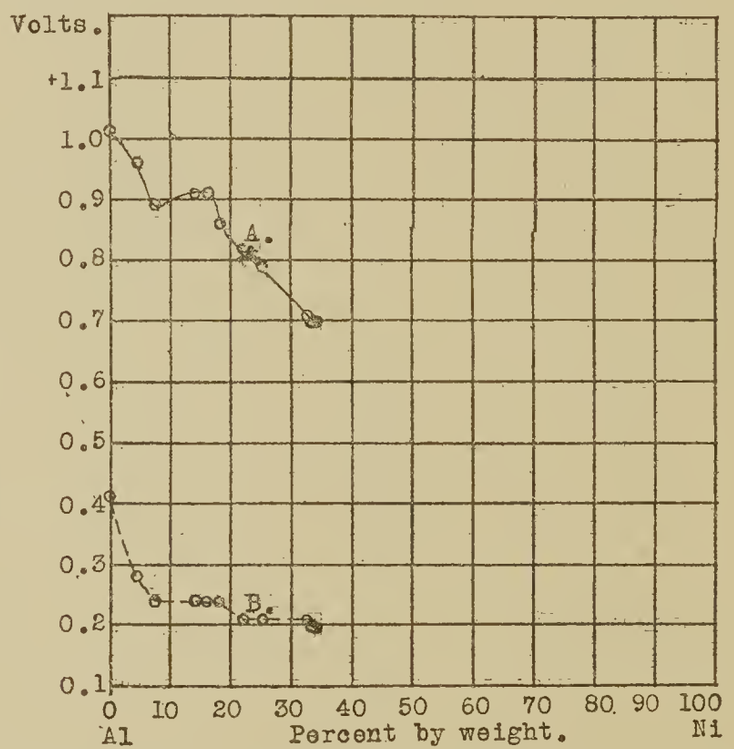

FIG. 12. Maximum Single Potentials of Al-Ni Alloys in *N* $AlCl_3$ Solution. A = Potentials of the Amalgamated Alloys. B = Potentials of the Unamalgamated Alloys.

The potential of zinc either when the metal is amalgamated or unamalgamated is practically the same. The results obtained for the copper amalgamated and unamalgamated were exactly the same, while the value for the amalgamated nickel was 0.04 volt smaller than for the unamalgamated nickel. The lower value in the case of the amalgamated nickel was perhaps due to an extra heavy coating of mercury; however, the measurements made upon pure nickel were not as constant as the measurements for pure zinc and pure copper. Of the four metals, nickel, copper, zinc and aluminum, it is the aluminum whose potential is changed to a noticeable degree by amalgamation. Since this is true, we would naturally expect the potentials of the aluminum alloys also to be changed considerably by amalgamation, as has actually been found to be the case.

2. Specific Gravities and Other Physical Properties. With the single exception of alloy number twelve, which contained 56 percent of nickel, all of the specific gravities were determined by the same method as that described in the section on aluminum-copper alloys. The method was that of weighing each alloy in the air and then suspending each under water by a silk fiber, thereby obtaining the loss in weight, and from these data calculate the specific gravities. Number twelve, being in a granulated form, could not be measured by the same method as the others. A sample of this alloy was thoroughly cleaned of grease by means of alcohol and ether. It was then weighed in air and then in an Ostwald pycnometer, the latter being filled with water. Before weighing the metal in the pycnometer, the air was removed by means of a pump. The specific gravities of the alloys are tabulated in Table IX, the curve being plotted in Fig. 13.

The alloys containing small percents of nickel are very tough, but above 10 percent of nickel this property rapidly disappears, the alloys becoming much harder and more brittle. With more than 25 percent of nickel, the alloys are very liable to crack when they are clamped in a vise sufficiently tight to permit their being sawed with a hack saw. As has already been stated, the 56 percent nickel alloy crumbled, without the influence of any external force, two weeks after it was cast. All of the alloys of nickel and aluminum that were prepared were non-magnetic.

Samples of the alloys numbers 2, 7, 10, and also of pure nickel, number 12, were subjected to corrosion tests in a 5 percent salt solution. Details of the method may be found in the section on aluminum-copper, under the heading "Physical Properties." The results of this test were that all of the samples corroded except the pure nickel; however, the initial corrosion of the amalgamated samples was greater than that of the samples which had not been amalgamated. It might be said for the amalgamated samples that the greater the percent of aluminum in the alloy, the greater the corrosion. The 56 percent nickel alloy of aluminum disintegrated after standing in the air two weeks without ever having been placed in the salt solution, whereas the 55 percent zinc alloy of aluminum and the 56 percent copper alloy of aluminum disintegrated as a result of being subjected to the action of the salt brine. It is peculiar how much alike these three

alloys of aluminum are, the percent of aluminum in each being about the same.

TABLE IX.

Single Potential and Specific Gravity Results of the Series.

Number of Alloy	Percent of Nickel	Single Potential Amalgamated Alloy Volts	Single Potential Unamalgamated Alloy Volts	Specific Gravity
1	0	1.01	0.41	2.70
2	4.55	0.96	0.28	2.75
3	7.51	0.89	0.24	2.82
4	14.25	0.91	0.24	2.95
5	16.31	0.91	0.24	3.00
6	18.21	0.86	0.24	3.05
7	22.22	0.82	0.21	3.15
8	25.43	0.79	0.21	3.27
9	32.93	0.71	0.21	3.54
10	33.69	0.70	0.20	3.60
11	34.16	0.70	0.20	3.65
12	56.00	*	*	5.16
13	100.00	—0.36	—0.32	8.90

* Not determined.

DISCUSSION AND CONCLUSION.

While an insufficient number of alloys have been prepared to draw any conclusions with regard to the form of the single potential curve for the entire series, nevertheless, from the data of Table IX it is quite evident that by amalgamation the initial potentials of the alloys which contain less than 35 percent of nickel are much higher than the potentials which are measured upon the unamalgamated specimens. Just as in the aluminum-copper series, there is a break in the curve for the readings made upon the amalgamated alloys of aluminum and nickel. The minimum occurs at 7 percent of nickel, while the eutectic, according to Gwyer, corresponds to 6.5 percent of nickel. It will be recalled that the break in the curve representing the readings taken upon the amalgamated alloys of the aluminum-copper series also occurred at about the eutectic for that series. The potential of the 56 percent nickel alloy of aluminum was not obtained, since it was not in a suitable condition to warrant any measurements being made.

The specific gravity curve plotted in Fig. 13 has been drawn by means of a heavy line for only that part of the series in which a number of alloys were produced, that is, for the alloys containing less than 35 percent of nickel. The remainder of the curve has been prolonged by means of a dotted line, since the specific gravities of only one alloy and pure nickel were obtained for the remainder of the series.

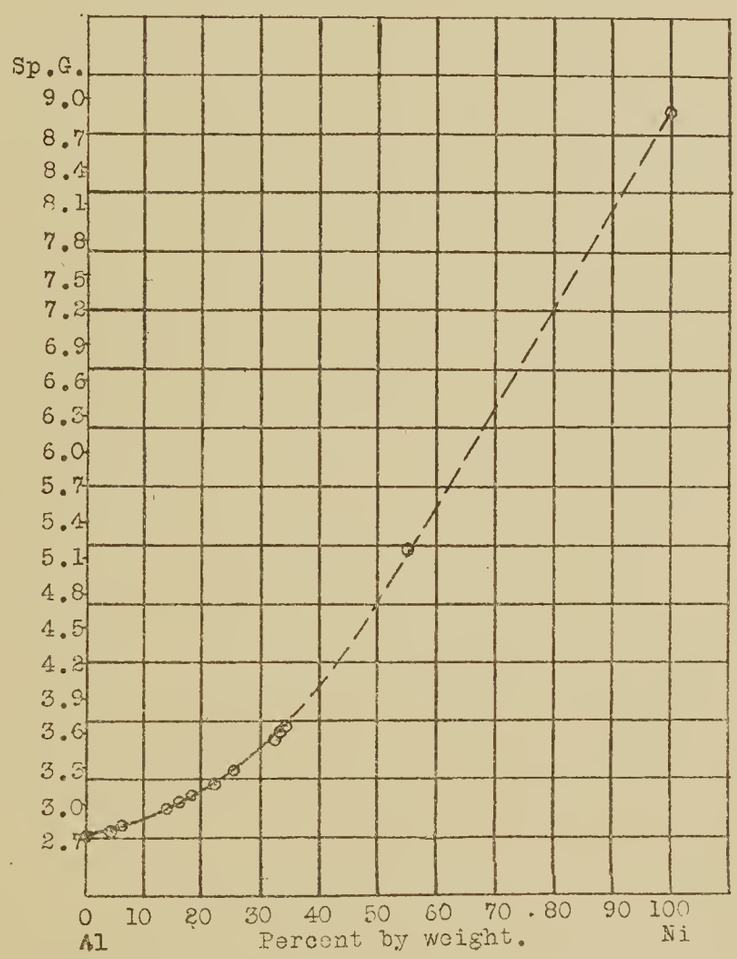

FIG. 13. Specific Gravities of Aluminum-Nickel Alloys.

With pure aluminum, and the alloys of aluminum with copper and aluminum with zinc, it was necessary to make the initial potential readings upon the amalgamated alloys within less than five seconds after placing the alloys in the aluminum chloride solution for measurement. From the data of Table VIII, which data are quite characteristic of the readings made upon the other alloys of this series, it is evident that the initial readings made upon the amalgamated specimens are not the maximum readings. The maximum readings were obtained within one minute after

immersing the amalgamated alloys in the aluminum chloride solution for measurement. We are not able to explain just why the maximum potentials measured for this series were not obtained just after immersing in the aluminum chloride solution instead of after thirty seconds. This same table also clearly shows that to obtain the maximum potential values, one should not wait for the specimen to reach an equilibrium. Thirty seconds after alloy number six, when amalgamated, had been placed in the aluminum chloride solution, it had reached a maximum value which, after standing ten minutes, had dropped almost to the value of this same alloy when unamalgamated.

The ideal method of producing alloys of aluminum, which have melting points above the limit of the gas furnace, would be by means of an electric furnace either in vacuum or in the presence of an inert gas. This would eliminate the difficulties of oxidation that one encounters when working with aluminum at high temperatures and in the presence of oxygen. Such furnaces were not available at this university, and consequently the remaining alloys of this series could not be prepared and studied at this time.

The authors wish to express their thanks to Professor O. P. Watts and Mr. E. O. Kraemer for many helpful suggestions as well as aid in carrying on this investigation.

University of Wisconsin,
Madison, July, 1919.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

MANGANIN[1]

By M. A. Hunter and J. W. Bacon.[2]

Abstract.

The authors measure the effect of small variations in the percentages of manganese, nickel and iron on the electrical resistivity and on the temperature coefficient of resistivity. They also studied its thermo-electromotive force against copper. They conclude that small variations in manganese affect the resistivity but not its temperature coefficient; that small quantities of iron affect the temperature coefficient considerably. Care must be taken in annealing the wire to avoid oxidation of the manganese in it.—[J. W. R.]

Since the importation of manganin from abroad has ceased, the American manufacturer has found considerable difficulty in producing material which will meet the exacting requirements of the trade.

While no definite specifications for this material have ever been written it has been generally understood that manganin is an alloy of copper, manganese and nickel, with an extremely low temperature coefficient of electrical resistance. The value of this coefficient cannot be definitely stated. It varies with slight variations in the composition of the wire. Thus Feussner and Lindeck[3] found on measuring 42 manganin resistances that

31 had co-efficients.....	1×10^{-5}
6 had co-efficients.....	2×10^{-5}
3 had co-efficients.....	3×10^{-5}
1 had co-efficient	3.3×10^{-5}
1 had co-efficient	8.1×10^{-5}

[1] Manuscript received March 17, 1919.

[2] Rensselaer Polytechnic Institute, Troy, N. Y.

[3] Feussner and Lindeck. Wissenschaftl. Abhandl. der Reichsanstalt. (1895), 2, 503.

Again Rosa[4] states that samples of manganin tested in the Bureau of Standards in 1909 gave between 15° C. and 30° C. a temperature coefficient varying between 4 and 20 parts in a million per degree but that it sometimes exceeded 20 parts per million. At another time[5] he states that "between 15° C. and 25° C. the mean value of this coefficient varies from 1 to 22 parts in a million per degree and sometimes goes as high as 40 or 50 parts per million. In general the temperature coefficient becomes negative at temperatures between 20° C. and 25° C. If, however, the temperature coefficient over the working range (15° C. to 30° C.) is kept below 10 parts per million, an uncertainty in the temperature of 0.1° C. introduces an error in the resistance of one part per million, and with care the uncertainty can be kept below this figure."

We may, therefore, assert that a manganin wire will be satisfactory as a resistance standard if its temperature coefficient of electrical resistance is less than 1×10^{-5} per degree C.

COMPOSITION.

The standard hand books give somewhat conflicting information on the composition of manganin. The composition indicated by Feussner and Lindeck[6] contained copper 84 percent, manganese 12 percent and nickel 4 percent. Pender[7] and Liddell[8] suggest compositions which are identical with, or approximating to this. The Standard Hand Book[9] (McGraw) gives as the composition of the material copper 65 percent, ferromanganese 30 percent and nickel 5 percent. This last material is made after the specifications in the original Weston patent. The resistivity of this material is indicated as varying between 41.4 and 73.8 microhm-cm. and the temperature coefficient is given as varying between 1.1 and 3.9×10^{-5} per degree C. at 20° C.

In view of this conflicting mass of information it is not surprising that the manufacturer finds himself at a loss for accurate information in initiating the production of the material.

[4] Rosa. Bull. Bureau Standards, 5, 419.

[5] Rosa. Bull. Bureau Standards, 5, 421.

[6] Lindeck. Report of the Electrical Standards Comm. British Assn., 1892. Appendix IV, page 143.

[7] Pender. Am. Hand Book for Elec. Eng. (1914), 195.

[8] Liddell. Met. and Chem. Hand Book (1916), 166.

[9] Standard Hand Book for Elec. Engineers. Section IV, 149.

The question becomes further complicated when we investigate the material sold as manganin which we find already on the market. A manganin bar[10] made from old German stock analyzed as follows:

Copper	82.62 percent
Manganese	12.82 percent
Nickel	3.78 percent
Iron	0.72 percent
Total	99.94 percent

The variations of the electrical resistance of this material were as follows:

Temp. ° C.	Resistance
14.	0.96382
18.	0.96380
24.	0.96375
28.	0.96370
31.	0.96365
39.	0.96352

These figures do not show the characteristic maximum between 20° C. and 25° C. The value of the temperature coefficient between 18° C. and 24° C. is 0.87×10^{-5} ohms per ohm-degree.

Another analysis of a manganin sample given by Liddell[11] is as follows:

Copper	82.12 percent
Manganese	15.02 percent
Nickel	2.29 percent
Iron	0.57 percent

No measurements of the resistivity or the temperature coefficient of resistance are given for this material.

For a complete account of the work done by Feussner and Lindeck on the variations in composition in manganin reference should be made to their original papers.[12]

It would appear that no investigations have been carried on with a view to determining the effect of small percentages of

[10] Private communication from The Leeds and Northrup Co.

[11] Liddell. Met. and Chem. Hand Book (1916), 482.

[12] Feussner and Lindeck. Metallegirungen für Elektrische Widerstände. Z. für Instr. Kunde (1889), 9, 233.
Wissenschaftl. Abhandl. der Reichsanstalt. (1895), 2, 503.
Neue Materialen für elektrische Messwiderstände, Elektroch. Zeit. (1892), 13, 99.
Uber den Leitungswiderstand der Legierungen von Nickel, und Kupfer. Verh. der physik. Gesellsch. zu Berlin (1892), 109.

impurities in the commercial wires. Since the presence of these impurities is inevitable, by reason of the fact that the constituents from which the wire is made are not in themselves pure, it seemed of interest to determine the effect of some of these variations.

In this present paper the effect of small variations in the percentage of manganese, nickel and iron is taken under consideration.

EXPERIMENTAL RESULTS.

Production of Materials.

Throughout the investigation we have confined ourselves to consideration of material made according to the approximate proportions given by Feussner and Lindeck.

The copper used was electrolytic copper. The nickel was shot nickel from the International Nickel Co., analyzing as follows:

Nickel	99.07 percent
Iron	0.46 percent
Carbon	0.11 percent
Sulphur	0.03 percent
Total	99.67 percent

The manganese was Goldschmidt manganese reduced by aluminum.

The materials were melted in a covered Dixon graphite crucible placed in an electric furnace. The melt was cast into a bar in an open mold. A piece was cut off and rolled cold and drawn to a wire of approximately 0.07 cm. diameter. The wire was then annealed after a method indicated in a later part of this paper.

The stock from which the wire was drawn was subsequently analyzed. The analyses are recorded in the summary of results. In all cases it was found that the manganese content in the material was always considerably less than the amount introduced; a fact which is to be attributed to the ready oxidizability of the manganese.

METHOD OF ELECTRICAL MEASUREMENTS.

The electrical measurements were made by taking simultaneous readings on two Wolff potentiometers No. 1 and No. 2. The same standard cell was used with each potentiometer; thus elimi-

nating temperature errors in the standard cell. In Fig. 1, *R* represents a one-ohm standard Wolff resistance of which the temperature coefficient was known and corrected for. *A* and *B* are slide-wire rheostats, *A* being of high resistance and used for fine adjustments. *C* is a storage battery, and *X* the unknown resistance or sample of manganin under test. Both *X* and *R* were mounted in oil baths, that of *X* being arranged with a coil for cooling and heating. All measurements on a given sample were made at the same current value. This was accomplished by setting potentiometer No. 1 at a given value and obtaining a

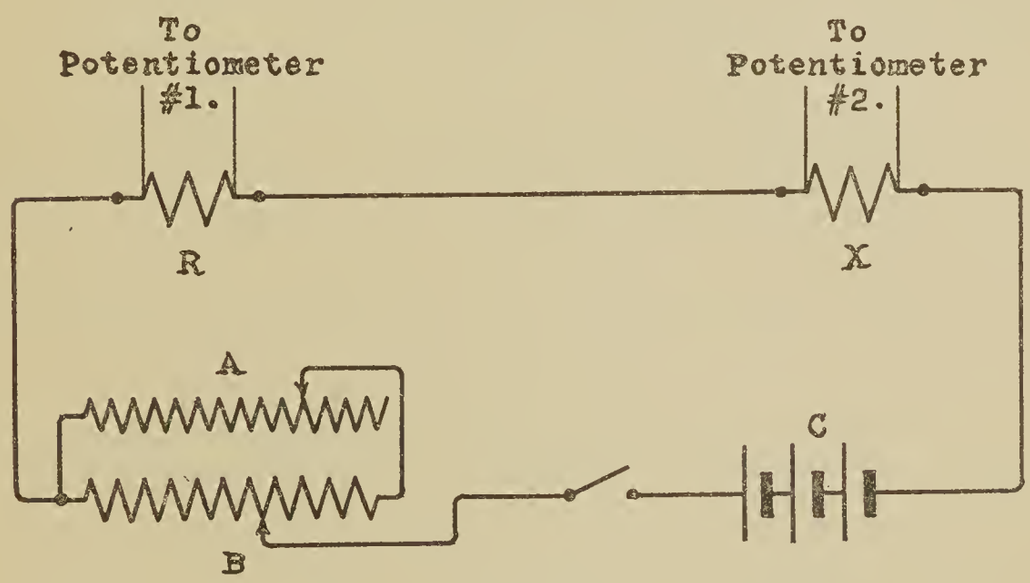

FIG. 1.

balance by varying *A* and *B*, a simultaneous reading being taken on potentiometer No. 2. The samples were mounted in an open spiral of about two centimeters diameter suspended from arms of the Wolff standard resistance pattern, with suitable potential leads. The thermometer for temperature observation was placed at the center of this spiral.

1. The first sample was made by melting together the materials in the following proportions:

Copper	86 percent
Manganese	12 percent
Nickel	2 percent

The electrical resistance of a wire made from this material was found to be as follows:

Material 10D.

Temperature Std. R. ° C.	Temperature X ° C.	Potentiometer No. 2 Volts	Resistance X Ohms	Resistance Reduced*
17.5	10.1	1.05117	0.875983	0.999832
17.7	16.0	1.05129	0.876083	0.999935
17.9	20.7	1.05135	0.876133	1.00000
18.1	25.9	1.05140	0.876183	1.00006
18.4	30.7	1.05142	0.876200	1.00008
18.6	34.8	1.05145	0.876225	1.00011
18.8	39.9	1.05143	0.876208	1.00009

* These resistances of the various wires have been calculated on the basis of one ohm at 20° C. and the results are plotted on the accompanying plate.

Potentiometer No. 1 volts held at 1.20000.
Mean diameter = 0.0721 cm. Length = 104.8 cm.

The resistivity of this material at 20° C. is 34.2 microhm-centimeters and the temperature coefficient is 1.1×10^{-5} ohms per ohm-degree between 18° C. and 24° C.

2. We next made up an alloy by melting together the following materials:

Cu	82.12 percent
Mn	15.02 percent
Ni	2.29 percent
Fe	0.57 percent
Total	100.00 percent

and obtained the following results:

Material 9D.

Temperature Std. R. ° C.	Temperature X ° C.	Potentiometer No. 2 Volts	Resistance X Ohms	Resistance Reduced*
21.2	10.5	1.15488	1.15494	0.999931
20.9	14.8	1.15494	1.15499	0.999974
20.6	20.1	1.15496	1.15501	0.999991
20.4	24.3	1.15498	1.15503	1.00001
20.3	27.2	1.15498	1.15503	1.00001
16.7	31.5	1.15493	1.15493	0.999922
16.7	35.4	1.15485	1.15485	0.999853
16.7	38.8	1.15480	1.15480	0.999810

* These resistances of the various wires have been calculated on the basis of one ohm at 20° C. and the results are plotted on the accompanying plate.

Potentiometer No. 1 volts held at 1.00000.
Mean diameter = 0.0714 cm. Length = 83.5 cm.

The last three readings in the above table were taken at a different time from the rest and are apparently in error. They do not, however, affect the deductions. The specific resistance of this material at 20° C. is 55.6 microhm-centimeters and the temperature coefficient is 0.33 x 10^{-5} ohms per ohm-degree between 18° C. and 24° C. The resistance-temperature curve shows a pronounced maximum between 20° C. and 23° C. This curve is radically different in this respect from the curve for 10D. We ascribe this difference to the presence of the small amount of iron in the melt.

3. In order to check this assumption we next made an alloy containing:

Cu	84.0 percent
Mn	13.5 percent
Ni	2.5 percent
Total	100.0 percent

with the following results:

Material 11D.

Temperature Std. R. ° C.	Temperature X ° C.	Potentiometer No. 2 Volts	Resistance X Ohms	Resistance Reduced*
13.1	10.1	1.04781	0.95253	0.99979
12.5	15.3	1.04796	0.95265	0.99991
13.4	16.0	1.04797	0.95266	0.99992
13.5	20.3	1.04805	0.95274	1.00001
13.7	25.3	1.04811	0.95280	1.00008
13.8	32.1	1.04820	0.95288	1.00016
13.9	35.8	1.04822	0.95290	1.00018
14.1	43.0	1.04824	0.95293	1.00020

* These resistances of the various wires have been calculated on the basis of one ohm at 20° C. and the results are plotted on the accompanying plate.

Potentiometer No. 1 volts held at 1.10000.
Mean diameter = 0.0713 cm. Length = 101.6 cm.

The resistivity of this alloy is 37.4 microhm-centimeters at 20° C. and the temperature coefficient is 1.6 x 10^{-5} ohms per ohm-degree between 18° C. and 24° C. The curve is identical in its trend with the curve for 10D in which iron was not present.

4. The next three melts were accordingly made with a small addition of iron as follows:

Material.

	12D	13D	14D
Cu	83.10	83.46	83.46
Mn	13.85	13.78	13.78
Ni	2.46	2.46	2.46
Fe	0.59	0.30	0.30
Total	100.00%	100.00%	100.00%

The results of resistance measurements on these samples were as follows:

Material 12D.

Temperature Std. R. ° C.	Temperature X ° C.	Potentiometer No. 2 Volts	Resistance X Ohms	Resistance Reduced*
17.9	10.5	1.14571	1.14571	0.999913
17.7	13.7	1.14577	1.14577	0.999965
17.5	19.9	1.14581	1.14581	1.00000
17.2	25.1	1.14582	1.14582	1.00000
17.1	29.5	1.14581	1.14581	1.00000
18.2	29.5	1.14578	1.14578	0.999887
18.3	35.5	1.14571	1.14571	0.999913
18.4	35.2	1.14571	1.14571	0.999913
18.5	38.5	1.14564	1.14564	0.999852

* These resistances of the various wires have been calculated on the basis of one ohm at 20° C. and the results are plotted on the accompanying plate.

Potentiometer No. 1 volts held at 1.00000.
Mean diameter = 0.0666 cm. Length = 83.8 cm.

Material 13D.

Temperature Std. R. ° C.	Temperature X ° C.	Potentiometer No. 2 Volts	Resistance X Ohms	Resistance Reduced*
22.3	10.7	1.25980	1.25988	0.999897
22.4	15.0	1.25987	1.25995	0.999952
22.4	18.0	1.25991	1.25999	0.999984
22.4	20.0	1.25993	1.26001	1.00000
22.5	22.4	1.25994	1.26002	1.00001
22.6	27.5	1.25993	1.26001	1.00000
22.6	30.0	1.25989	1.25997	0.999968
22.7	35.7	1.25985	1.25993	0.999936
22.8	39.7	1.25977	1.25985	0.999873
22.7	41.1	1.25975	1.25983	0.999863

* These resistances of the various wires have been calculated on the basis of one ohm at 20° C. and the results are plotted on the accompanying plate.

Potentiometer No. 1 volts held at 1.00000.
Mean diameter = 0.0714 cm. Length = 99.4 cm.

Material 14D.

Temperature Std. R. ° C.	Temperature X ° C.	Potentiometer No. 2 Volts	Resistance X Ohms	Resistance Reduced*
21.5	11.1	1.24625	1.24631	0.999904
21.6	14.7	1.24631	1.24637	0.999952
21.7	20.7	1.24637	1.24643	1.00000
21.6	24.5	1.24639	1.24645	1.00002
21.6	28.5	1.24640	1.24646	1.00002
21.5	33.8	1.24633	1.24639	0.999968
21.5	38.7	1.24628	1.24634	0.999928

* These resistances of the various wires have been calculated on the basis of one ohm at 20° C. and the results are plotted on the accompanying plate.

Potentiometer No. 1 volts held at 1.00000.
Mean diameter = 0.0669 cm. Length = 85.7 cm.

SUMMARY.

The data presented in the preceding tables are plotted on Plates No. 1 and No. 2.

A summary of the results is given in the following table:

Sample	Analysis of Material				Temp. Coeff. 18°-24° C.	Resistivity in Microhm-cms. at 20° C.
	Cu	Mn	Ni	Fe		
10D	88.02	9.93	1.74	0.24	1.2×10^{-5}	34.2×10^{-6}
11D	87.24	10.26	1.77	0.52	1.5×10^{-5}	37.4×10^{-6}
9D	88.20	8.84	1.78	0.93	0.33×10^{-5}	55.6×10^{-6}
12D	83.60	12.03	3.41	1.04	0.22×10^{-5}	47.8×10^{-6}
13D	84.72	12.83	2.08	0.73	0.38×10^{-5}	50.8×10^{-6}
14D	84.07	12.98	2.60	0.82	0.57×10^{-5}	51.1×10^{-6}

From the above table the following conclusions may be drawn:

The specific resistance except in the case of 9D increases with the content of manganese in the wire.

The temperature coefficient of electrical resistance is closely related to the iron content of the wire. In the first two samples 10D and 11D, to which no iron was added (the amount present being due to the original impurities present in the materials used) the temperature coefficient is high and the maximum resistance is found to be around 40° C. With increasing additions of iron the temperature coefficient becomes progressively lower and the maximum resistance is reached at lower temperatures. The best material No. 12D contains around 1 percent of iron. The maxi-

mum resistance is found at 23° C. The temperature coefficient is further very low indeed.

The percentage of nickel appears to bear no direct relation to either the resistivity or the temperature coefficient of the material.

In this connection it may be mentioned that Lindeck found that a wire made to contain copper 84, manganese 12, and nickel 4, had its maximum resistance at 45° C. and gave between 18° and 24° a temperature coefficient of 1.5×10^{-5}. In the light of the information here disclosed these two facts are explained by the probable low content of iron in the materials used.

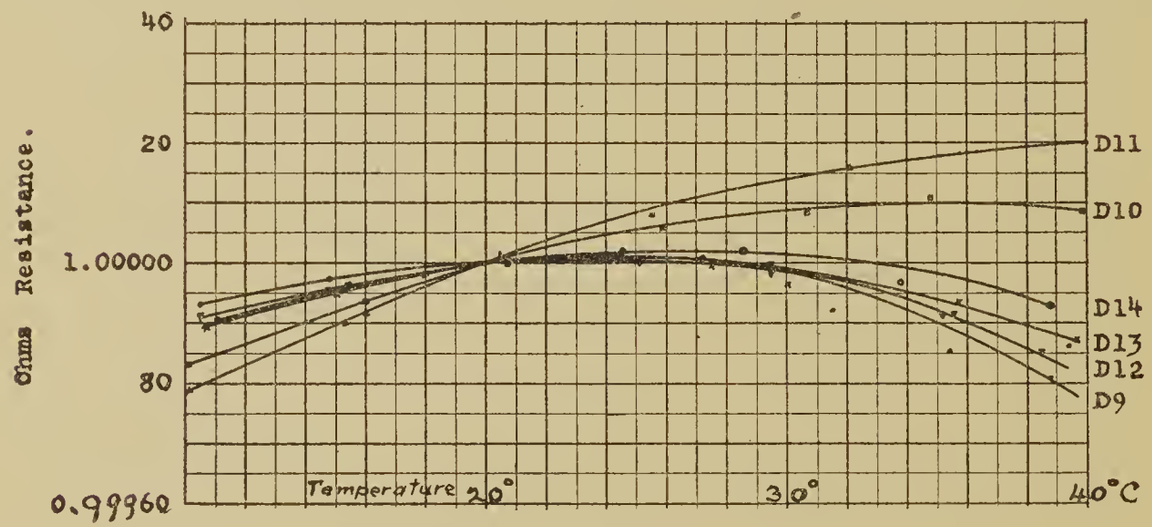

PLATE 1.

In general then it may be said that the resistivity of a manganin wire may be varied by changing the amount of manganese in the wire, but that a low temperature coefficient with maximum resistance within the working ranges can be obtained only by having an iron content around 1 percent in the wire.

THERMO-ELECTROMOTIVE FORCE AGAINST COPPER.

The following measurements of thermo-electromotive force were obtained in the various samples. The measurements were taken between 0° C. and 50° C.

Wire	E. M. F.
9D	.....3 microvolts per °C.
10D	.....4 microvolts per °C.
11D	.....5 microvolts per °C.
12D	.....8 microvolts per °C.
13D	.....4 microvolts per °C.

In all cases the thermo-electromotive force against copper is low.

ANNEALING THE WIRE.

In the annealing of a manganin wire there are two points of prime importance which have to be considered.

It has already been pointed out by Feussner and Lindeck that if a manganin wire be annealed at a low temperature in air, the

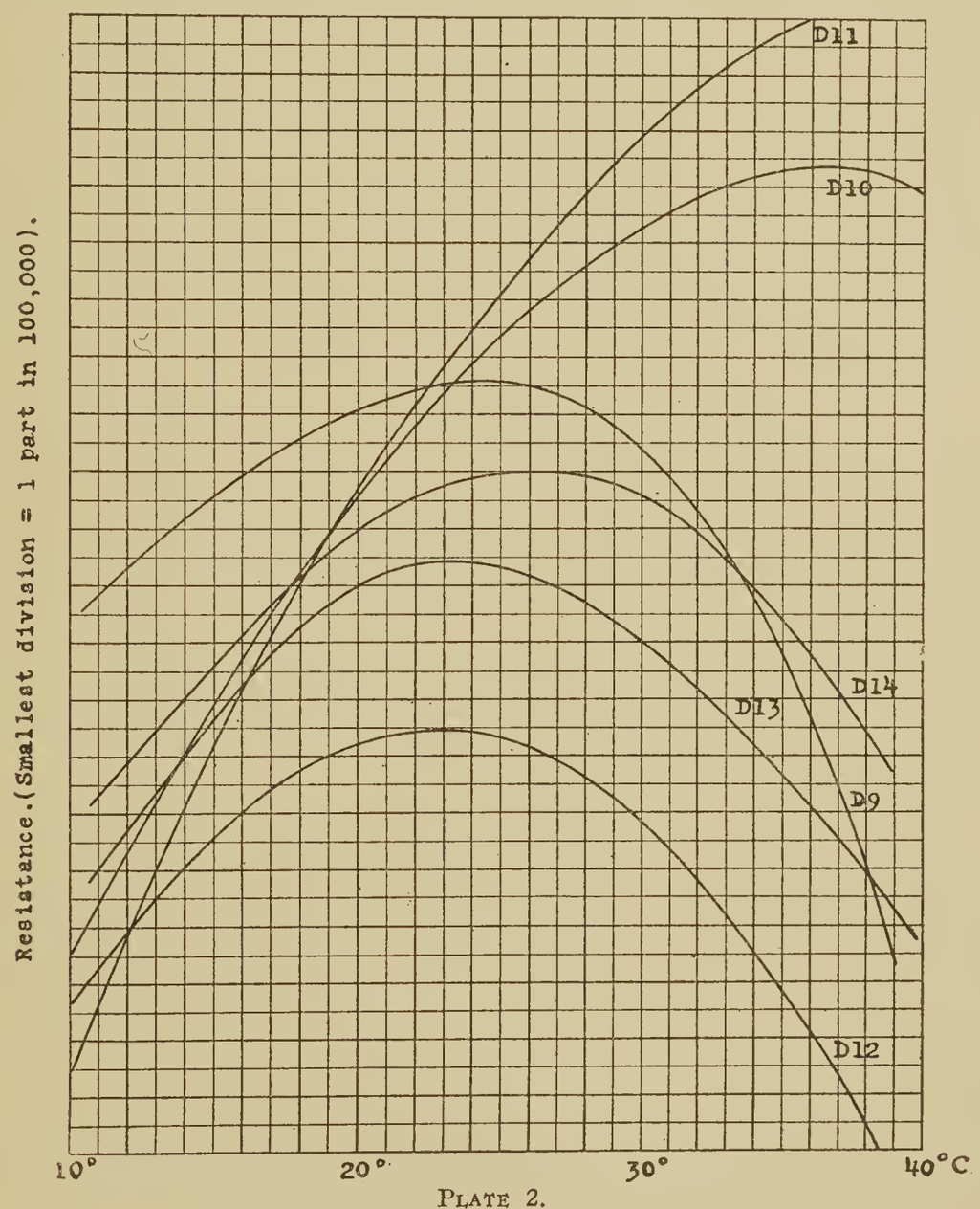

PLATE 2.

manganese in the wire suffers a selective oxidation. The result of this selective oxidation is that a layer of metallic copper is left coating the wire. Under these conditions the wire will give a high positive temperature coefficient, the value depending on the thickness of the coating.

In our experiments this observation has been confirmed. To

avoid this oxidation we found it necessary to anneal the wire in a current of carbon dioxide. Even then if the wire showed any bloom of oxidation on its surface, this had to be removed with fine emery paper before the manganin characteristics could be obtained.

Again Feussner and Lindeck have indicated the necessity of a long anneal at a low temperature to prevent subsequent variations in the resistance in a given length of the wire. This observation we have also confirmed.

To obviate these difficulties it is sufficient to anneal the wires at 700° C. for a few minutes in a stream of carbon dioxide and then maintain them at 150° C. in an oil bath for five hours before any measurements are made.

CONCLUSIONS.

We have not attempted in this investigation to vary the component metals in the manganin alloy over any wide range.

From the results obtained, however, these facts stand out as worthy of notice.

1. The percentage of manganese between the limits used affects the resistivity of the wire but has no effect on the temperature coefficient of resistance.

2. The presence of iron affects the temperature coefficient to a considerable degree. Those wires in which the iron content was low did not show the temperature coefficient reversal which is characteristic of a manganin wire. The results show that the presence of iron up to 1 percent improves the temperature coefficient of the resulting alloy.

3. We have confirmed the observation that during the annealing of the wire scrupulous care must be observed to avoid its oxidation.

4. We have further confirmed the observation that in order to stabilize the wire it is sufficient to anneal it at 150° C. in an oil bath for a period of 5 hours.

This research was suggested by the Leeds and Northrup Co. We desire in concluding to thank them for the aid they have

rendered us throughout the work. We must also thank Dr. A. T. Lincoln of this Institute for undertaking the labor of the chemical analyses.

Russell Sage Laboratory,
Rensselaer Polytechnic Institute,
Troy, N. Y.

DISCUSSION.

CARL HERING[1]: May I suggest that the term "iron" in the abstract be explained to be an impurity, because iron, as I understand it, is not one of the primary ingredients in manganin, and yet the abstract of the paper gives that impression.

J. W. RICHARDS[2]: That may be a fact, but it is seldom free from iron, and if some of the formulæ for making it by using ferro-manganese are followed out, you will have iron as an essential constituent. The Standard Hand Book of the McGraw Publishing Company gives in a recipe the use of ferro-manganese, so you would have iron if you followed this prescription in making it.

CARL HERING: I was referring only to the prescription given in the paper.

F. E. CARTER[3]: One and one-half percent iron is actually added as a primary constituent.

M. A. HUNTER (*Communicated*): The presence of iron in commercial manganin is undoubtedly due to the impurities in the manganese from which the material is made. The authors show that one percent of iron has a beneficial effect in the direction of shifting the maximum on the resistance curve nearer to the room temperature. If the component metals from which the alloy is made do not contain one percent of iron, it is suggested that enough be added to raise the amount to one percent, in order to obtain the beneficial result above mentioned.

[1] Consulting Electrical Engineer, Philadelphia.
[2] Professor of Metallurgy, Lehigh University.
[3] Physical Metallurgist, Baker & Co., Inc., Newark, N. J.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

COMMERCIAL POSSIBILITIES IN THE ELECTROCHEMICAL PRODUCTION OF ORGANIC COMPOUNDS.[1]

By C. J. Thatcher.[2]

We all know that, in industrial applications at least, the electrochemistry of the organic compounds has not kept pace with the progress in other lines of electrochemical activity. Probably most of us would have difficulty in naming more than a few organic chemicals which have been successfully manufactured by electrochemical methods, at least in this country. Indeed, progress in this direction has been so restricted that there has been, or seems to be, hardly any interest in this branch of the electrochemical industry.

It is true that in February, 1915, the New York section of this Society held a symposium consisting of three papers on the subject of organic electrochemical manufacturing, followed by a discussion in which a number of members took part. Both optimistic and pessimistic opinions regarding the future were expressed in the papers and discussions. Whatever may have been the general consensus of opinion, it is certain that the symposium did not cause any noticeably increased activity in the electrochemical production of the carbon compounds, despite the apparently favorable opportunity presented by the war. At that time I was myself preparing to manufacture para-amido phenol sulphate—an intermediate and photographic developer—electrochemically; but no other serious attempt to establish organic electrochemical operations during the war now ended, has come to my attention.

Perhaps the period intervening since that symposium has not been the most propitious; for old and established processes were almost certain to bring needed and highly profitable results, and there was little or no time for laboratory or plant experimentation.

[1] Manuscript received Sept. 26, 1919.

[2] Consulting Chemist, New York City.

However, that period is now passed, and enterprising American manufacturers are again confronted with the necessity for improvement and economy in production, in order that they may keep ahead of domestic and foreign competition.

Since it is in the new, and comparatively favored field of dyestuff and other organic products, that industrial preparedness against foreign competition will be most necessary, it would seem that we electrochemists should now seriously consider, with an open mind, whether we cannot blaze a path in this, as we have in other directions. We do not need to be reminded that Yankee optimism, courage and ingenuity have, when once aroused, turned failures into successes, and confounded the wise in other countries. Our enemies—in war as in chemistry—have had surprises along that line in the Argonne and elsewhere.

Now we have not really tried to manufacture organic derivatives here, electrochemically. There is much that we do not know about, of what has been done or tried in other countries, particularly in Germany. But the mere fact—if it be a fact—that others have tried and failed, does not conclusively prove anything as to what the American electrochemist can do.

The real question, which we ought to consider in all seriousness, is, whether now, and in this country, is not the time and place to begin more or less extensive industrial applications of organic electrochemistry. To awaken some sustained interest, consideration and discussion of this question, in this or other meetings, is the object of my paper. And for this purpose I would ask you to consider, first, the basic principles which may indicate the kind of electrochemical operation which could be expected to be successful commercially; and, thereafter, to consider briefly specific electrochemical processes.

To this end we may inquire first as to how economic values are created by industrial electrochemistry. That is a question which Dr. Roeber considered in his presidential address before this Society in April, 1914,[3] and it seems well to apply some of Dr. Roeber's considerations to the subject of this paper.

Economic values are created by industrial electrochemistry, as in other similar lines of activity, either as time value, place value, or form value. Time or place value of organic electrochemical

[3] Trans. Am. Electrochem. Soc. (1914), **25**, 17.

products is mainly due to their high energy content, which can be imparted to them more economically by the electric current. Such products are valuable mainly because they enable us to transmit their stored energy to another time or place. Calcium carbide and aluminium when used in alumino-thermic reactions are examples of electrochemical products having a high time and place value. Their potential energy becomes kinetic whenever or wherever desired. These and other products of electrochemical industries acquire their economic value by serving as a medium for chemical power transmission. Many or most of this class of products are made by electrothermic methods.

But another and important class of electrochemical products has nothing to do with chemical power transmission. These are products which have a high form value, *i. e.*, those which derive their intrinsic value from the mechanical and chemical work expended and dissipated in transforming the raw materials into the desired finished product. Qualities, other than a high specific content of energy, impart the economic value to this class of compounds. Caustic soda, bleaching powder, and electrolytically refined copper are examples. Such products are made by electrolysis, for the most part; they have a high form value due to the work expended during electrolysis, upon comparatively cheap raw material. In the production of nitric acid or nitrates by atmospheric fixation, the raw material—air—is more than cheap, it has no economic value, we may say. Therefore, the form value of nitric acid and nitrates so produced is unusually high.

On the basis of past experience, and reasoning by analogy, we may conclude, therefore, that products which have a high form value are such as stand the best chance of being economically produced by electrolytic methods. The comparatively wide margin between the value of the raw material and the finished product permits a considerable saving whenever electrolytic methods reduce the cost of the mechanical and chemical work put into the operation. This comparatively large saving often more than offsets the interest and other charges due to the design and construction of electrochemical equipment.

This somewhat abstruse consideration has a very practical bearing upon the subject of this paper, for it will help to decide whether electrochemical methods of production may be expected

to be economical in the manufacture of organic compounds, and it should enable us to tackle the problem along the lines of least resistance. Electrothermic methods are almost, but not quite, excluded in the manufacture of the carbon compounds. It is in the technical applications of electrolysis that electrochemical operations should succeed if at all. And our prior consideration teaches that a very large number of organic compounds should be capable of being produced electrochemically, because of the vast number of organic compounds which have a high form value. The carbon compounds (particularly the aromatic compounds, such as dyestuffs), have a high form value, more so than inorganic compounds as a class. The average high value of such compounds is not due to a high energy content, nor to the value of the organic raw material, which is often comparatively low. Their value is due, rather, to the amount of manual, mechanical and chemical energy expended and dissipated in transforming the relatively cheap raw material or intermediate into pure, finished product. And we have seen that just such transformations are a most promising field for industrial electrolytic operations.

Advantages Which May Inhere in Electrochemical Methods.

Obviously, in the final analysis, electrochemical methods are advantageous only when they are more economical. But economy may be due to several causes. We will consider only the production of known products already in demand, and now produced by chemical methods, and not new compounds which the characteristics of the electrode processes might enable us to produce.

First. Electrochemical methods may supplant chemical methods when the same organic raw material is used

(a) By increasing the yield through control of conditions to be later considered.

(b) By eliminating inorganic raw materials such as oxidizing or reducing agents, as by substituting electrolytically generated oxygen and hydrogen.

(c) By electrolytic regeneration of inorganic raw materials, as for example, spent chromic acid liquors.

Calculation will show that with but few exceptions, it is cheaper to oxidize, reduce, or substitute by electrolytic than by chemical

means—a fact which was brought out by F. A. Lidbury in the symposium previously referred to.[4]

This is particularly true when, as is sometimes possible, both the anode and cathode reactions may be made to perform useful work, either in the preparation of the same or different organic products.

But even without such possible economy, electrochemical transformations figured on an equivalent basis, are usually surprisingly cheaper. For example, with power at metropolitan rates, it costs about ten times as much to oxidize with bichromate as at the anode of a well-designed cell. And if an electrolytic oxidation saves 35 cents per pound, buying power at metropolitan rates, it is not necessary and may not pay to locate the plant in an inaccessible locality in order to use hydro-electric power at an additional saving of only a few cents per pound.

Whenever, therefore, electrolytic oxygen, hydrogen, chlorine, etc., give as good a yield per unit of organic raw material, we have every reason to expect economy. For any increased charges, inherent in the use of electrolytic equipment, such as interest and depreciation, need not, if the cell is properly designed, be enough to nearly eliminate the economy in favor of the electrolytic method.

There are numerous organic compounds now prepared chemically by oxidation, reduction or substitution, which, by skillful control of conditions in a properly designed cell, can be more economically made electrochemically from the same raw materials as those now used, for one or more of the reasons specified above. Only poor yields, excessive depreciation, etc., can prevent success, and it will be shown that improvement in these particulars can be effected.

Second. Electrochemical methods may supplant chemical methods when a cheaper organic raw material may be used.

It would carry us beyond the scope of this paper to consider the various instances in which such a substitution may be made. Briefly stated, the conditions obtaining at the surface of an electrode are so much more variable and controllable than in purely chemical transformations, so that new reactions can be used technically, or a number of intermediate steps may be combined into one operation.

[4] Met. and Chem. Eng. (1915), 13, 211

One of the features of electrode processes which accounts for the economy thereby effected, is the wide range of gas pressure obtainable at the surface of an electrode by suitable variation of the electrode potential. Referring to this point, Prof. Nernst stated[5] that it was possible to liberate a gas, such as chlorine, at the anode either in greater than homeopathic dilution, or at pressures which amount to millions of atmospheres, and that this vast range of pressure made it possible to readily chlorinate organic compounds to every conceivable degree. The importance of the regulation of gas pressure in organic synthesis has been shown in many hundreds of instances. Thus, in organic substitution processes, the number of substituted atoms is often dependent upon the pressure at which the reaction occurs. Since any desired pressure can be so conveniently created, and maintained at an electrode, solely by variation in electrode potential, it is obvious that electrochemical methods should frequently be more direct and economical. As Professor Haber once stated: "Whereas the nucleus of the aromatic compound is not attacked by most chemical oxidizing materials, or is completely destroyed, accompanied by the disruption of the ring, electrolysis under favorable conditions permits a smooth oxidation with the result that the hydroxyl group is introduced into the benzol nucleus. Benzol is thus oxidized in sulphuric acid solution at the anode to hydroquinone, aniline to para-amido-phenol, azo-benzol to tetra-oxybenzol, anthracene to alizarine."

No explanation of the economy resulting from such direct syntheses of coal tar crudes is needed.

Third. Electrochemical methods may supplant chemical methods when a high degree of purity is desirable.

We can oxidize or reduce by electrical energy without contaminating the finished product by foreign materials, the complete removal of which is generally troublesome and expensive.

The three general considerations above outlined should be sufficient to show why it will be advantageous and economical to use electrochemical methods in manufacturing organic compounds. And we have also seen that the use of such methods may be expected to be successful in the manufacture of products having a high form value. Let us consider the other side briefly.

[5] Berichte 1897, p. 1562.

Disadvantages Which May Inhere in Electrochemical Methods.

The objections to the commercial use of electrochemical methods in preparing organic compounds were very concisely stated at the symposium in 1915, by F. A. Lidbury, in the following words: "In the first place, to do a unit of chemical work in a unit of time, the electrolytic plant would be usually very much larger than a chemical plant; require very much more room; be usually very much more expensive; require more expert attention, and on account of the extreme severity of the demands of electrolytic methods on materials employed for plant, etc., the upkeep would usually be higher.

"In the second place, the working out of an electrolytic method requires a very much greater amount of time, thought, intelligence and expenditure than the working out of a chemical method. Though this might not amount to much as regards laboratory investigation, anyone acquainted with the electrochemical industry would be aware of the greater difficulties which invariably have to be overcome in preparing such a process for large scale operation, and in that important phase known as 'established practice.' "

I am not sure that we can all agree as to the validity of the first of these objections. However, is it not a fact that such objections could be raised against the adoption of any electrolytic process, inorganic as well as organic? It would seem that almost any electrochemical plant is necessarily larger and is more expensive to design, construct, run and maintain. We cannot deny the commercial practicability of organic electrolytic plants upon grounds which would deny the practicability of existing, successful electrolytic plants. Wherever, for reasons already given, there is any considerable economy of operation in electrolytic methods, higher interest charges due to greater expense in the design, equipment, and maintenance of an electrolytic plant, are more than offset, at least in any plant producing annually 100,000 pounds or more of an organic product.

Conditions Favoring Economy in Operation.

Electrochemical methods of production are, of course, only economical when operated under skillfuly selected and controlled conditions. It is not my purpose to consider such conditions at

any great length in this paper. They include electrode material, chemical and electrical conditions, catalytic effects and diaphragm material. Excepting the latter two, the complexity and range of variation is too great to be considered advantageously in any general manner. But it will be worth while to consider briefly factors capable of a more general consideration such as catalytic effects and the use and nature of diaphragms.

Catalytic Effects.

The nature and importance of catalytic action upon electrode processes were set forth by Prof. Ostwald with customary insight, in his paper presented at the International Electrical Congress at St. Louis in 1904.[6] A few quotations from this paper will be most instructive. Prof. Ostwald stated: "If there are several possibilities for the reaction at the electrode, the readily occurring chemical reaction depends not only on the nature of the possible chemical reactions, but upon the concentration of the substances which are present, and by suitably varying the latter we can bring any reaction to any point in the voltage series.

"In connection with this it should be emphasized that what is called briefly *'the electrode process'* is in reality a rather complex phenomenon. * * * * It will thus be seen that even the simplest electrode reactions are made up of a plurality of reactions following each other step by step. Since each of the same can be influenced catalytically, it is already evident that a very great variety of results is possible. * * * *

"Such means of changing the speed of a given reaction within very wide limits, exists really in chemistry in what is called *catalyzers*. Herefrom it is at once evident which very important and essential part the catalyzers are able to play in electrolytic action. By retarding one of the possible reactions by means of a negative or retarding catalyzer, we are able to *exclude* its influence from the electrolytic process. By accelerating by means of a catalyzer a reaction which is of itself slow, we can *include* it into the electrolytic process. In other words we are no longer compelled to let the current act in the way stated by the above simple law, but in principle, we are now able, *by applying suitable cata-*

[6] Met. and Chem. Eng. (1904), 2, 393.

lyzers, to prescribe to the current that reaction which we want to take place, and it does not matter at which point in the voltage series this reaction is situated.

"This fundamental idea may be applied in two directions. First, in synthetic work; if we suppress by negative catalyzers those reactions which would take place before the desired one, we can bring the desired reaction to the front, or, if the reactions which take place before the desired one take place with a low speed, we can accelerate the speed of the desired reaction by means of a positive catalyzer and can thus produce the same effect. The second application is *for analyzing the reactions which really take place at the electrodes.*"

The improvements which may be effected in industrial organic syntheses by suitable control of catalytic action are sufficiently outlined for present purposes in the foregoing. A more detailed consideration of the subject will be found in Prof. Ostwald's paper, in President Bancroft's recent address on "Poisoning of Catalytic Agents,"[7] and in papers[8] describing my investigations in Prof. Ostwald's laboratories which called his attention to the subject.

Use and Nature of Diaphragms.

It is probable that the need of suitable diaphragms for an electrolytic cell for the manufacture of organic compounds, has hindered the development of the industry as much or more than any other factor. On account of the nature of organic compounds, many of which are either insoluble or non-electrolytes, it is usually necessary to employ a solvent electrolyte, which tends to disintegrate diaphragms so rapidly, as to prohibit commercial operations. Few materials suitable for diaphragms will withstand the corrosive effect of strong acids, alkalis, or oxidizing liquids for any length of time. Other materials which might be more resistant become clogged during use, accompanied by a prohibitive increase in resistance.

I encountered such difficulties at the outset of my own organic electrochemical manufacturing, and was obliged to devise a diaphragm suitable for the purpose. This diaphragm, known as

[7] Trans. Amer. Electrochem. Soc. (1917), **32**, 459-462.

[8] Zeit. Phys. Chem., **45**, 225-230; (1909), **47**, 687. Met and Chem. Eng. (1904), 2, 452-454.

"Electro-Filtros,"[9] has been in commercial use in organic electrochemical operations since 1915; and experience has shown that it is absolutely permanent in acid and neutral solutions and adapted to the purpose. It is not suitable for use in strong alkalis; but organic electrolytic operations involving the use of any other kind of solvent, need not be unsuccessful through lack of a suitable diaphragm.

Some Suggested Organic Electrosyntheses.

Approximately 150 patents have been granted, most of them prior to 1910, for electrochemical processes producing organic compounds. The bulk of these are German patents, which fact alone tends to indicate the interest in Germany in this line. A list of a few of these and other suggested organic compounds will be sufficient for the purpose of this paper; but in preparing the list it has not been restricted to preparations of known or proven commercial practicability.

With this understanding it might be said that the following offer some possibilities: Alcohols, aldehydes and ketones of fatty acids, iodoform, vanillin, chloral, azo and hydrazo compounds, oxydation products of fusel oils, dyestuffs of the triphenylmethane type, aniline blue, aniline black, Hofmann's violet, anthraquinone, alizarine, congo red, orange II, sulfo acids, piperidine, dihydroquinoline, benzidine amido-phenol, chlor-benzols, quinone, hydroquinone, saccharine and phthalic acid.

Status of the Industry.

The technology of organic electrochemistry has, of course, reached its greatest development in Germany, as a natural consequence of Germany's attention to the organic field. The great secrecy maintained there in this line has made it impossible to obtain exact knowledge regarding the status of the industry. It has been stated on good authority that chloral, vanillin, iodoform, azo and hydrazo compounds have been profitably manufactured electrochemically and that one concern at least, was, prior to the war, operating a large electrochemical plant devoted to the production of anthraquinone.

[9] Met. and Chem. Eng. (1915), **13**, 336-38.

In Switzerland, according to my information, at least one electrolytic plant has been engaged in the production of chloroform, chloral, and phosphorous chlorides, and has been utilizing electrolytic chlorine in the manufacture of synthetic indigo.

If there have been any extensive developments in this country they have escaped my attention, excepting the use of electrolytic chlorine for the indirect production of mono-chlor-benzol and di-chlor-benzol and similar organic compounds. I have myself made a few tons of photographic developers (para-amido-phenol) electrochemically in the last few years and have produced hydroquinone and anthraquinone on a small factory scale.

One of the greatest obstacles to the progress of the industry has been the skepticism of manufacturers and financiers regarding the possibilities. That, however, is nothing unusual for it was not many years ago that the manufacture of steel in the electric furnace was ridiculed by both manufacturers and technologists. The next ten years or so will witness a development of the organic electrochemical industry which will surprise many, for, as has been shown in this paper, the underlying economic principles are favorable.

DISCUSSION.

COLIN G. FINK[1]: Four or five years ago we had a symposium in New York on the electrochemical production of organic compounds, and at that time it seemed almost impossible to get anybody to talk on the subject, or to find anybody who knew anything about it. A number of our members were good enough to go over the literature and present papers on what was known at that time. At the end of the symposium it was remarkable how many men came forward and said they had been working along these lines for some time but were not in a position to say anything. Among others, Dr. Thatcher had been working for a number of years on the production of photographic developers, and in the paper he gives us today we find a very clear account of the difficulties he encountered and suggestions as to remedies for the future, such as control of current density, control of temperature, the selection of proper solutions, etc. Dr. Thatcher himself

[1] Head of Research Laboratories, Chile Exploration Co., New York City.

is head of a concern manufacturing organic chemicals by electrochemical methods on a factory scale. These chemicals have been manufactured electrochemically by the Germans for years, but details of the processes were never published, and accordingly Dr. Thatcher and his assistants had to go carefully over a lot of ground and work out the process for each product step by step.

W. D. BANCROFT[2]: I can quite see that it might be economical if you can use both the chemical and anode reactions to produce useful substances, but as I remember, there was quite a discussion in Germany some years ago as to what the possibility was of finding useful reactions which could be run simultaneously, one with the anode and one with the cathode. That was a good deal more of a problem than anything else. I wondered how Mr. Thatcher succeeded in solving that.

COLIN G. FINK: Dr. Thatcher does not mention any specific instance; he just refers to that in a general way.

W. D. BANCROFT: You will have, of course, the higher voltage at the electrode where you are not getting a useful product, as there will be lack of polarization and you are losing one of your two hypothetical products. It seems to me a statement based on the assumption that you can get useful products at both anode and cathode is ambiguous, unless such reactions occur.

COLIN G. FINK: I suppose he can separate his anode and cathode compartments. Dr. Huff mentioned before lunch that practically all of the iodoform is now produced electrochemically. It has been produced electrochemically abroad for a number of years, but it is new for this country.

S. A. TUCKER[3]: Is it not true that we have a possibility of better control of oxidation and reduction due to the electrolytic method than by other means, and is it not also the fact that they have not only not applied the electrolytic method, but have not learned as much as they ought to about it? It seems to me that there are great possibilities here. I believe it is one of the most important lines of development for the future.

[2] Professor of Physical Chemistry, Cornell University.
[3] Chief Chemist, Chemical Foundation, Inc., New York City.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

RADIANT RESISTOR FURNACE.[1]

By F. A. J. FitzGerald.[2]

Abstract.

The author describes a radiant resistor furnace for the distillation of low-grade or scrap zinc, which was built and operated at the FitzGerald Laboratories and produced several tons of refined zinc. The best results were obtained with a current of approximately 845 A. at 65 volts or 55 kw. With this power the output was about 50 kg. refined zinc per hour.

In 1905 a small laboratory furnace in which the charge was heated by means of a radiant resistor was described.[3] A note about a furnace heated in a similar manner, but having an electrical capacity of 150 kw. was published in 1910,[4] and the description of another design appeared under the title of "A New Electric Resistance Furnace," in a paper given to the American Electrochemical Society in 1911.[5] This also had a capacity of 150 kilowatts, and, like the furnace described in 1910, was designed by Thomson and FitzGerald, the object in each case being the smelting of zinc ores by the Imbert process. The furnace described in 1911 was used in experimental work and was found to be very unsatisfactory as shown in a paper, "Note on an Unsuccessful Furnace Experiment," presented to the American Electrochemical Society in 1911,[6] but the experiments showed very clearly that the trouble was due to faulty design and construction, more particularly very imperfect heat insulation.

[1] Manuscript received May 5, 1919.
[2] Electric Furnace Expert, Niagara Falls, N. Y.
[3] Electrochemical and Metallurgical Industry, III, 218.
[4] Metallurgical and Chemical Engineering, VIII, 317.
[5] Transactions (1911), **19**, 273, *et seq.*
[6] Transactions (1911), **20**, 281, *et seq.*

In 1915 the cost of high-grade zinc increased so much that experiments on the distillation of low-grade or scrap zinc appeared to be worth while, therefore, Thomson, with our collaboration, designed a furnace on principles similar to those mentioned above and this was built and operated at the FitzGerald Laboratories, several tons of refined zinc being produced.

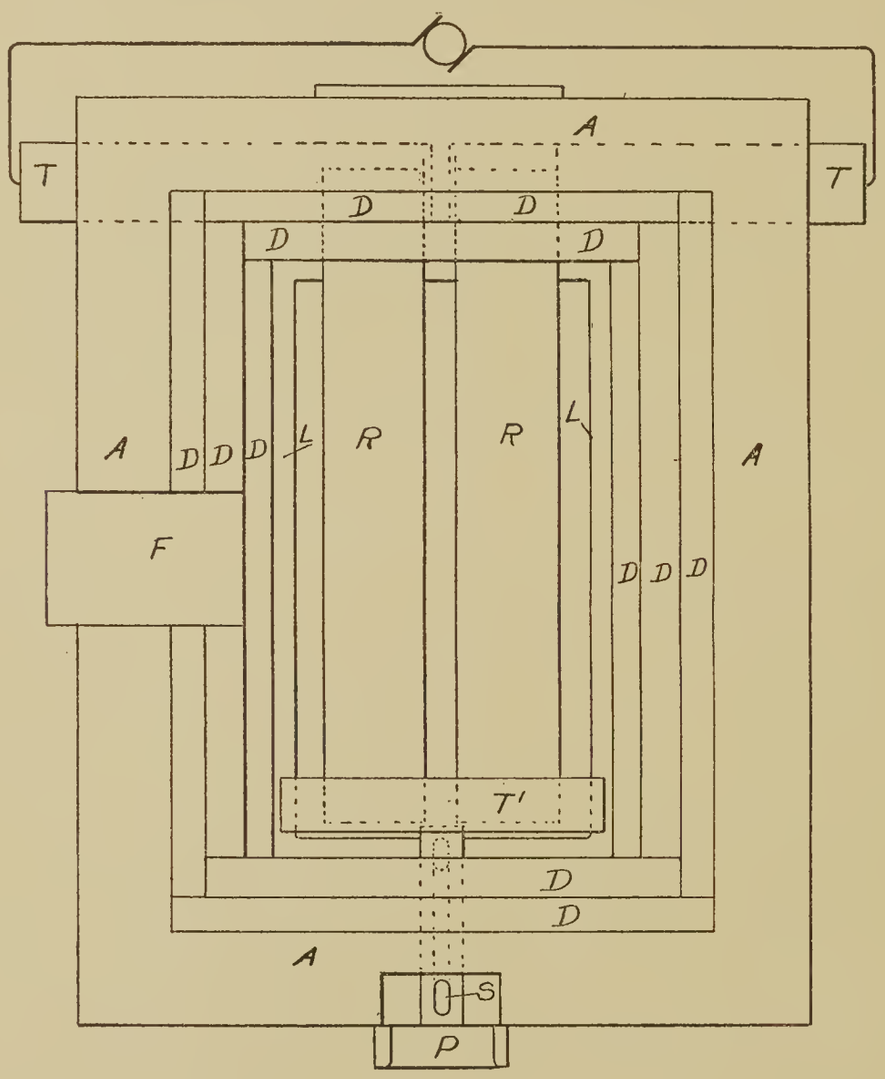

FIG. 1.

The diagrams of Figs. 1, 2 and 3 show the construction of the furnace; Fig. 1 with the cover removed, Fig. 2 a longitudinal vertical section, and Fig. 3 a transverse vertical section. The furnace was approximately 2 meters long (6½ feet) and the diagrams are drawn approximately to scale. The same lettering is used in all figures.

The parts of the furnace constructed of firebrick are marked *D*, and the heat insulation, which consisted of either Sil-O-Cel

or the insulating brick made by the Armstrong Cork Co., is marked *A*. The furnace hearth containing the charge was made of various materials, *L*, but the most satisfactory was a carbonaceous refractory called "Vitricarbo" manufactured by the National Carbon Company. The furnace was heated by two resistors *R* which were made from graphite slabs which had slots cut into them so as greatly to increase the path of the current. The current is brought to the resistors by the graphite terminals *T T* and the resistors are connected in series by the graphite connector T^1. The furnace was charged with molten zinc by means

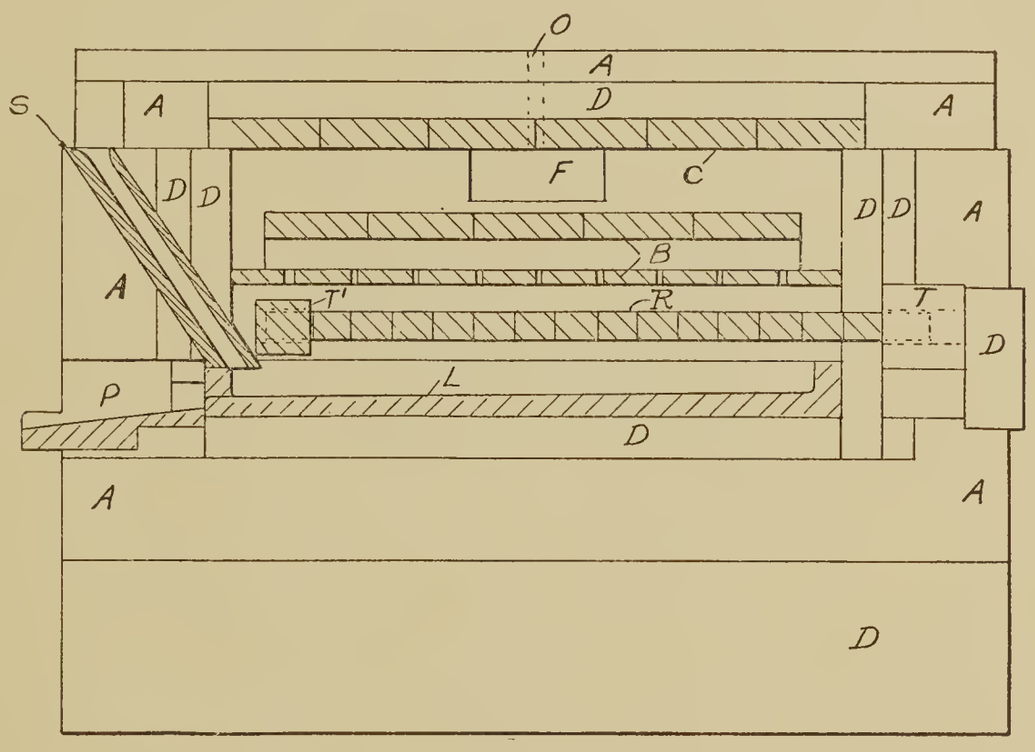

FIG. 2.

of the inclined tube *S*, the lower end of which was normally below the surface of the molten bath. Above the resistor were two diaphragms *B*. The lower one of these consisted of a set of plates laid transversely in the furnace, each plate being one inch distant from its neighbors, thus forming a perforated diaphragm. The upper diaphragm was made of two-inch (5 cm.) carbon slabs, and it did not extend to the ends of the furnace. Thus the zinc vapors had to pass up first through the openings in the lower diaphragm and thence flowed out round the ends of the upper diaphragm finally reaching the opening *F* which led to the condenser. The cover of the furnace was formed of carbon slabs

C on top of which were the firebrick *D* and finally the heat insulating brick *A*. At *O* there was a hole in the cover into which a pyrometer tube was inserted for measuring the temperature of the zinc vapors passing into the condenser through *F*. The condenser is not shown in the diagram, but was in the form of a flat box about 100 x 50 x 7 cm. with a series of transverse baffle plates. This was provided with means for preheating, at first electrically but subsequently with a charcoal fire, since once distillation was fairly under way external heating was unnecessary.

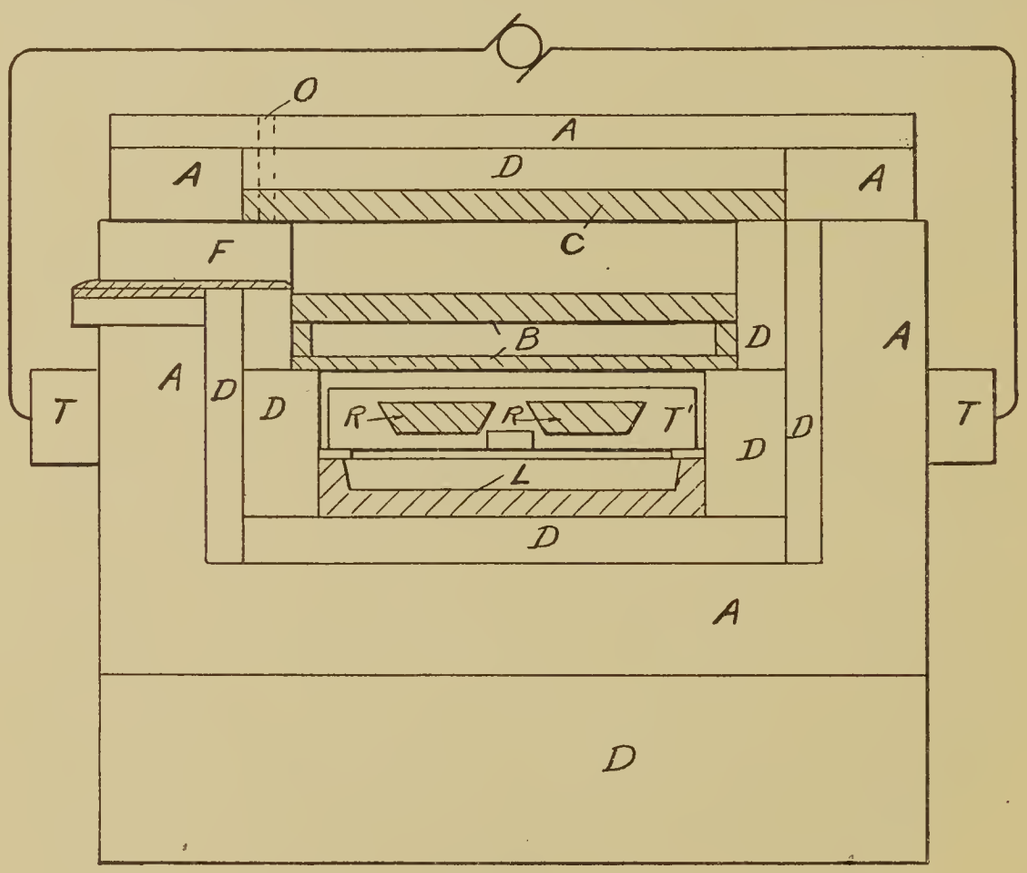

FIG. 3.

The furnace was originally designed with the idea that it would use 75 kw. or 1,000 amperes at 75 volts, and turn out 45 kg. of zinc per hour. It was found, however, that the best results were obtained with a current of approximately 845 amperes at 65 volts or 55 kw., and that with this power the output of distilled zinc was about 50 kg. per hour.

From previous experimental work with zinc furnaces much experience had been gained in the problem of condensing zinc vapor, so that no difficulty was found in designing a condenser which would give liquid zinc without the production of any blue

powder. There were plenty of other troubles with the condensers, mainly due to the difficulty of constructing the particular form used in such a way that it would not develop leaks. These difficulties were, however, overcome.

The method of working the furnace was as follows: When building it, before putting in the resistors, as much scrap or low-grade zinc was put into the hearth *L* as it could contain, and the construction of the furnace then finished. After starting, the current was at first kept very small so as to dry out the furnace slowly, then it was raised and more zinc from the premelter introduced through *S* until the lower end of *S* was well closed. Meanwhile the condenser was heated to a temperature slightly above the melting point of zinc. When the zinc vapor began distilling the temperature in the condenser rose till finally it was no longer necessary to supply external heat, but instead some of the heat insulation was removed. Thereafter it was merely necessary to keep a watch on the condenser temperature and slightly adjust the air-cooling devices. The zinc was tapped from the condenser at intervals, and premelted zinc introduced so as to keep the lower end of the tube *S* closed. From time to time the residue in the furnace, consisting mainly of zinc very high in iron and lead, was tapped from the furnace at *P*.

One interesting accident which happened once or twice was the plugging up of the charging tube *S* caused by pouring in molten zinc too near its freezing point. This was cleared by making electrical contact with the bath through the tap hole at *P* and then arcing on the frozen zinc with a rod introduced through *S*.

The experimental work on the distillation of zinc ran over many months, for all sorts of investigations were made on forms of resistors, designs of terminals, various kinds of premelting furnaces, small-scale experiments on fractional distillation of zinc alloys, production of blue powder, various forms of condensers, refractory materials, heat insulators, etc. But to give an idea of the furnace's working under normal conditions, a run of 37 hours may be taken, because this will show the behavior of the furnace after starting up and also what may be expected from it with continuous running under constant conditions. Energy

measurements were made at the furnace. The zinc charged into it was in the molten state, hence the figures for energy consumption do not include melting. Here are the data obtained:

Length of run	37 hours
Average power used during distillation	55.2 kw.
Total energy for distillation	2051 kw. hours
Minimum temperature of zinc entering condenser	1040 °C.
Average temperature of zinc entering condenser	1225 °C.
Average temperature of zinc in premelter	550 °C.
Weight of zinc condensed	1558 kg. (3494 pounds)
Weight of zinc condensed per hour	43 kg. (95 pounds)
Energy per kg. of zinc	1.29 kw. hours
Energy per pound of zinc	0.59 kw. hours

In order to get a notion of the behavior of the furnace when it has reached normal working conditions the last 9 hours of the run may be taken.

Length of period	8.9 hours
Average power used during distillation	54.4 kw.
Total energy for distillation	484 kw. hours
Av. temperature of zinc vapor entering condenser	1250 °C.
Average temperature of zinc in premelter	550 °C.
Weight of zinc condensed	455 kg. (1001 pounds)
Weight of zinc condensed per hour	51 kg. (112 pounds)
Energy per kg. of zinc	1.06 kw. hours
Energy per pound of zinc	0.48 kw. hours

A question that will naturally be asked is how much energy was used in premelting the zinc fed into the furnace. Unfortunately the records do not give a direct answer to this question, for much trouble was experienced with the premelter during the earlier part of the run. However, it is possible to get a fairly close approximation of what energy we used for premelting during the last 9 hours of the run from the following data.

Weight of zinc melted	968 kg. (2130 pounds)
Energy consumed for melting	216 kw. hours
Energy per kg. of zinc	0.22 kw. hours
Energy per pound of zinc	0.10 kw. hours
Weight of zinc transferred from melter to furnace,	632 kg. (1390 pounds)
Energy used to melt 632 kg. of zinc	139 kw. hours

Now, by referring to the figures given above for the last part of the distillation it is found that the total energy used in producing 455 kg. (1,001 pounds) of distilled zinc was 623 kw. hours, hence:

Total energy per kg. of zinc.....1.37 kw. hours
Total energy per pound of zinc.....0.62 kw. hours

It will be observed from these figures that the percentage of zinc refined by distillation in this particular experiment was only 72 percent of the amount charged, therefore, the amount of zinc which had to be premelted was greater than would have been the case had the distillation been carried further.

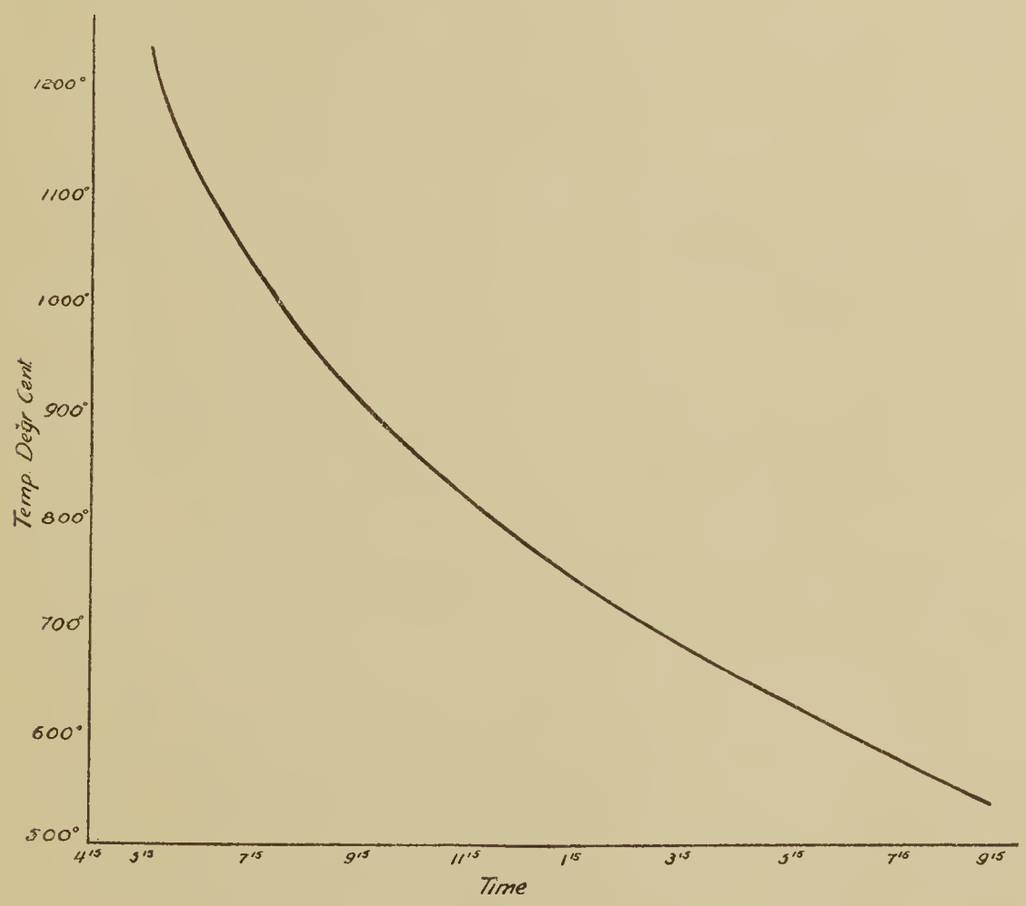

FIG. 4.

The curve shown in Fig. 4 is a cooling curve of the furnace.

As regards furnace troubles, the only one that amounted to anything was leaking of the hearth. This was simply due to faulty construction of the lining, a fault that was corrected by experience.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

ELECTRIC HEAT IN THE TYPEWRITER INDUSTRY.[1]

By A. M. Clark.[2]

Abstract.

An electrically heated oven is described for baking japan on various parts of typewriters. The method of use is explained and comparative tables show the better economy and larger capacity of the electrically heated oven, compared with gas or oil heated ovens.

The Royal Typewriter Co., of Hartford, Conn., is one of two companies manufacturing typewriters who bake the finish on typewriter parts in electrically heated ovens in preference to other methods now in use, such as in gas or oil heated ovens. In the following paragraphs will be found a description of the process involved, and the advantages which have resulted from the use of an installation of japan baking ovens equipped with electric heaters and control.

This installation consists of a battery of six ovens. Four of these ovens are 7 ft. 5 in. (2.2 m.) high, 7 ft. 1 in. (2.1 m.) deep, and 6 ft. 2 in. (1.9 m.) wide, while the other two are slightly smaller. Each oven has a connected load of 27 KW. The accompanying photographs show the battery of six ovens and the inside of one oven partially loaded with typewriter parts.

The heating units which rest on the floor take up very little space and run at a relatively low temperature. This low temperature, combined with the ventilation which is provided, practically eliminates the danger from explosions and fires. The ovens run almost without attention, as automatic control and a time clock insure the desired temperature and the throwing off of the current at a predetermined time.

[1] Manuscript received June 7, 1919.

[2] Industrial Heating Dept., General Electric Co., Schenectady, N. Y.

Oven No. 1 is used for baking japan on the spacer bar of typewriters. These spacer bars are made of soft wood, and are given five coats of a rubber japan, which gives a hard-rubber-like surface which cannot be easily dented. It was found advisable to use a temperature of 250° F. (121° C.) for 3 hours for the first four bakes. The last bake is run at a temperature of 275° F. (135° C.) for the same period of time.

The other five ovens are used for baking japan on the frames, ribbon spools, base plates, top dust plates, back dust plates, front

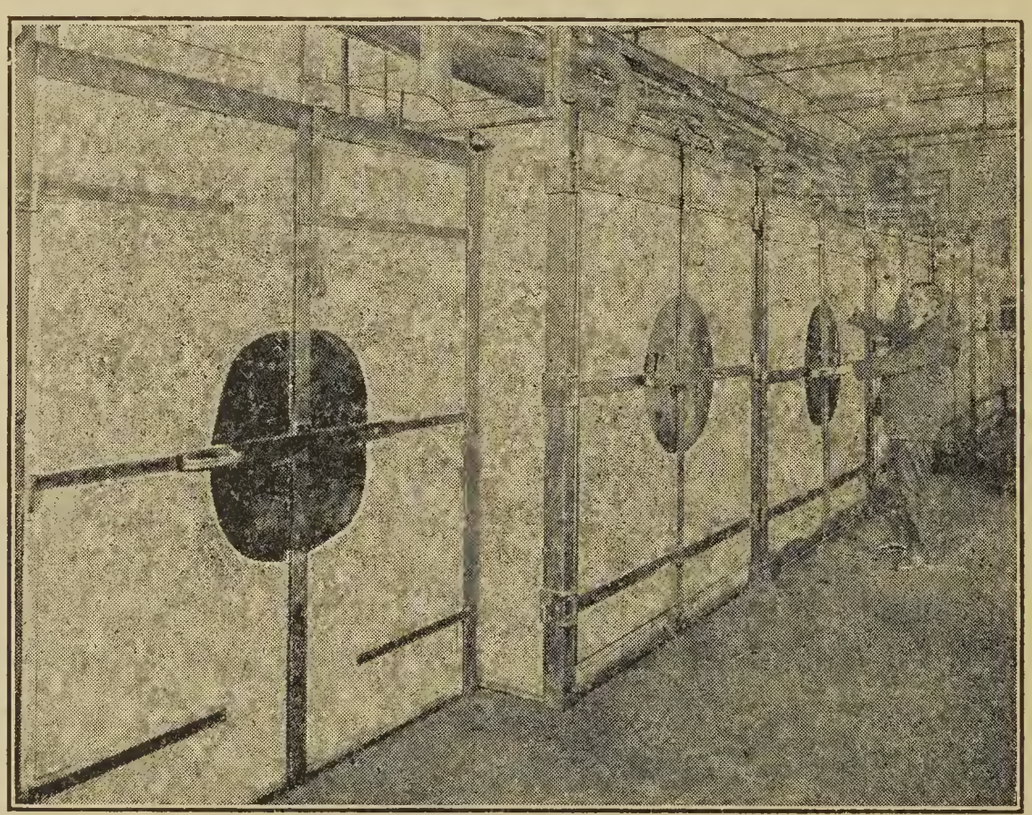

Fig. 1.

plates, and paper tables. The finish on these parts requires a more complicated system of baking and finishing. In the first of these ovens the parts to be baked, having first been dipped in a metal japan are baked for five hours at 350° F. (177° C.), after which they are rubbed down by hand and again put through the same process in the second oven. Before being placed in the third oven they are sprayed with a black-baking japan and baked for five hours at 400° F. (204° C.). After this bake the parts are all sanded and rubbed clean and the same operation is repeated using the fourth oven. They are then rubbed down by hand with a hard piece of felt and powdered pumice, giving them a very

smooth dull finish, sprayed with the same black-baking rubbing japan and baked for five hours at 400° F., when they are ready for the final assembly of the typewriter.

These ovens have been in operation for more than four years and have proven very satisfactory.

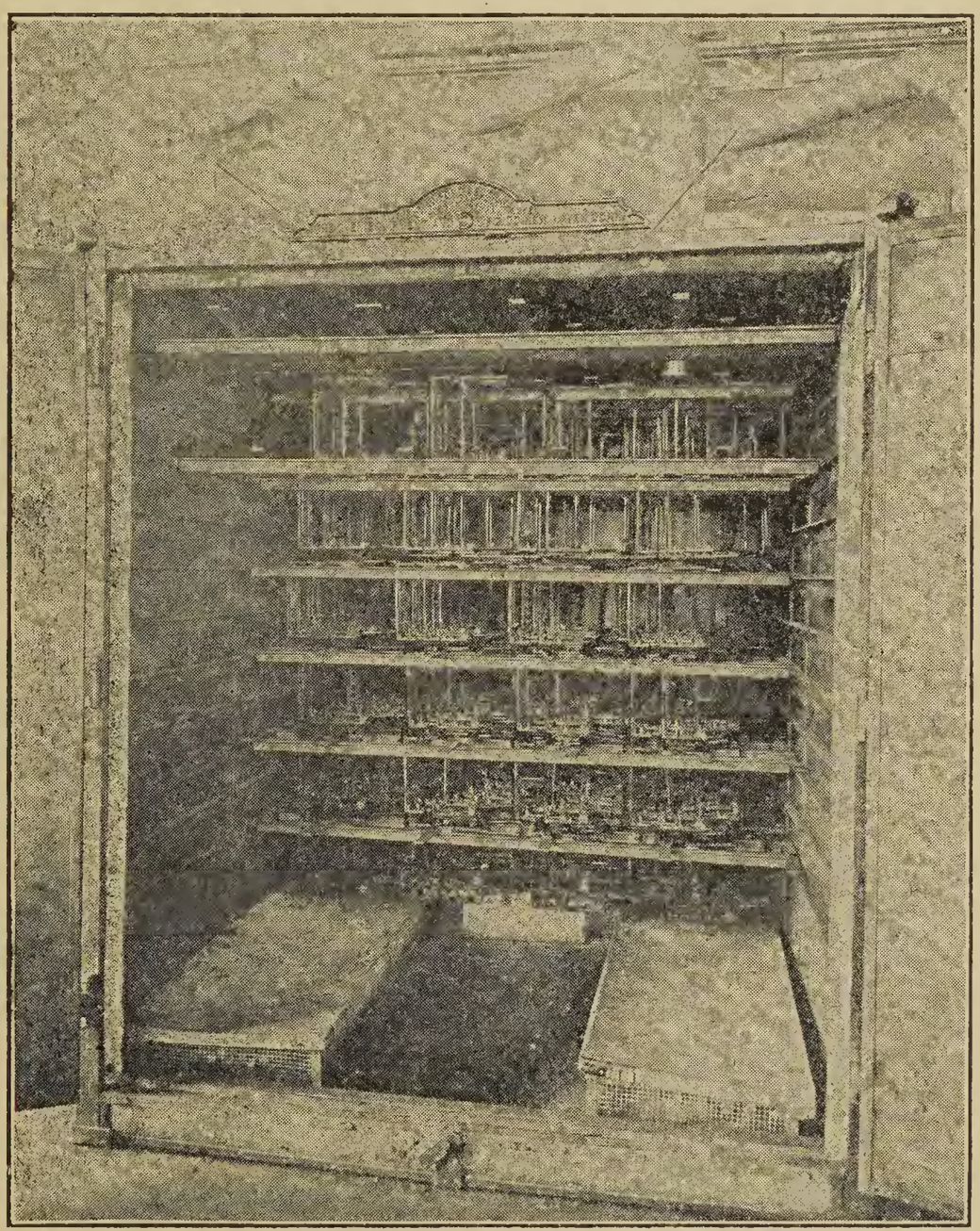

FIG. 2.

The following tables show the comparative capacity and cost of operation of two of the ovens, one equipped with gas and the other with electric heating equipment. The second table shows the comparative cost of gas and electricity per day, per month,

and per year, and a saving per year of more than 20 percent, or $360.36 for each oven.

SUMMARY OF FUEL COSTS PER BAKE IN JAPAN OVENS.

	Electricity	Gas
Capacity	300 frames	180 frames
Capacity	3,288 lb. (1,495 kg.)	2,055 lb. (934 kg.)
Fuel used per bake	123 K. W. H.	1,100 cu. ft. (31.5 cu. m.)
Rate	$0.015 less 40%	$0.85—12%=$0.75
Cost per bake	$1.10	$0.825
Cost per frame	$0.00366	$0.00458
Cost per pound	$0.000334	$0.000401

FUEL COST—FULL TIME

Cost per day	$ 4.62	$ 5.78
Cost per month	120.12	150.15
Cost per year	1,441.44	1,801.80
Saving per year	360.36	

or 20% of present costs, without allowance for savings in defective bakes.

From the above report it is evident that the Royal Typewriter Co. under the conditions existing in Hartford, Conn., is able to effect a considerable saving in dollars and cents by using electrically heated ovens. A superior finish without any increase in the cost of labor is also an item which makes the electrically heated ovens desirable.

In addition to the above, the Royal Typewriter Co. has found that electric heat proved advantageous in other ways as well. With gas-heated ovens their experience showed that owing to burning, underbaking, dust, and variations in the heat, a certain percentage of bakes was lost. It was also found that with electric heat they get a superior and uniform finish, which is durable and does not easily chip off.

There are a number of companies manufacturing typewriters who are undoubtedly contemplating the installation of electrically heated ovens to bake the finish on typewriter parts. As this method is economical and insures a very good and uniform finish, it should not be long before the typewriter industry is using electrically heated ovens exclusively.

General Electric Co.,
Schenectady, N. Y.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

ELECTRIC FURNACE FOR EXPERIMENTAL WORK[1]

By F. A. J. FitzGerald[2] and Grant C. Moyer.[2]

In our work we have found that for a variety of small-scale experiments the furnace which is shown in the drawings illustrating this note and which we use in the laboratory is convenient and satisfactory.

Fig. 1 is an end view of the furnace; Fig. 2, a side view; Fig. 3, a plan, in section on the line *aa* in Fig. 2; Fig. 4, details of the electrode holders.

The furnace is mounted on a truck built of channel irons, a pipe frame-work and boiler plate. The electrode holders and their working mechanism are mounted on the boiler plate and the furnace is built on this. The electrode holders are aluminum bronze castings and are moved to and fro by means of right and left-handed screws respectively. The shaft of each screw terminates in a gear engaging with gears mounted on a shaft which passes under the boiler plate base and which has a hand wheel mounted at one end. By this hand wheel one worker can attend to the running of the furnace. The hand wheel shaft is in two parts connected with a coupling and near this is a small collar on the shaft. This arrangement is used to form what we call a "gear lock" which consists of an iron bar held between the collar and the coupling. This bar is hinged and can be dropped down so that the hand wheel shaft may be pushed longitudinally in its bearings sufficiently far to throw the gears out of mesh. This allows the electrode holders to be separately adjusted when desired. The water cooling rings are bronze castings and slide on the electrodes. They are hollow and open at the top so that they can be kept filled with water, thus cooling the electrodes.

[1] Manuscript received May 21, 1919.

[2] The FitzGerald Laboratories, Inc., Niagara Falls, N. Y.

The rings should not be in the position shown in the drawing; but should be pushed up close to where the electrodes pass through the furnace walls.

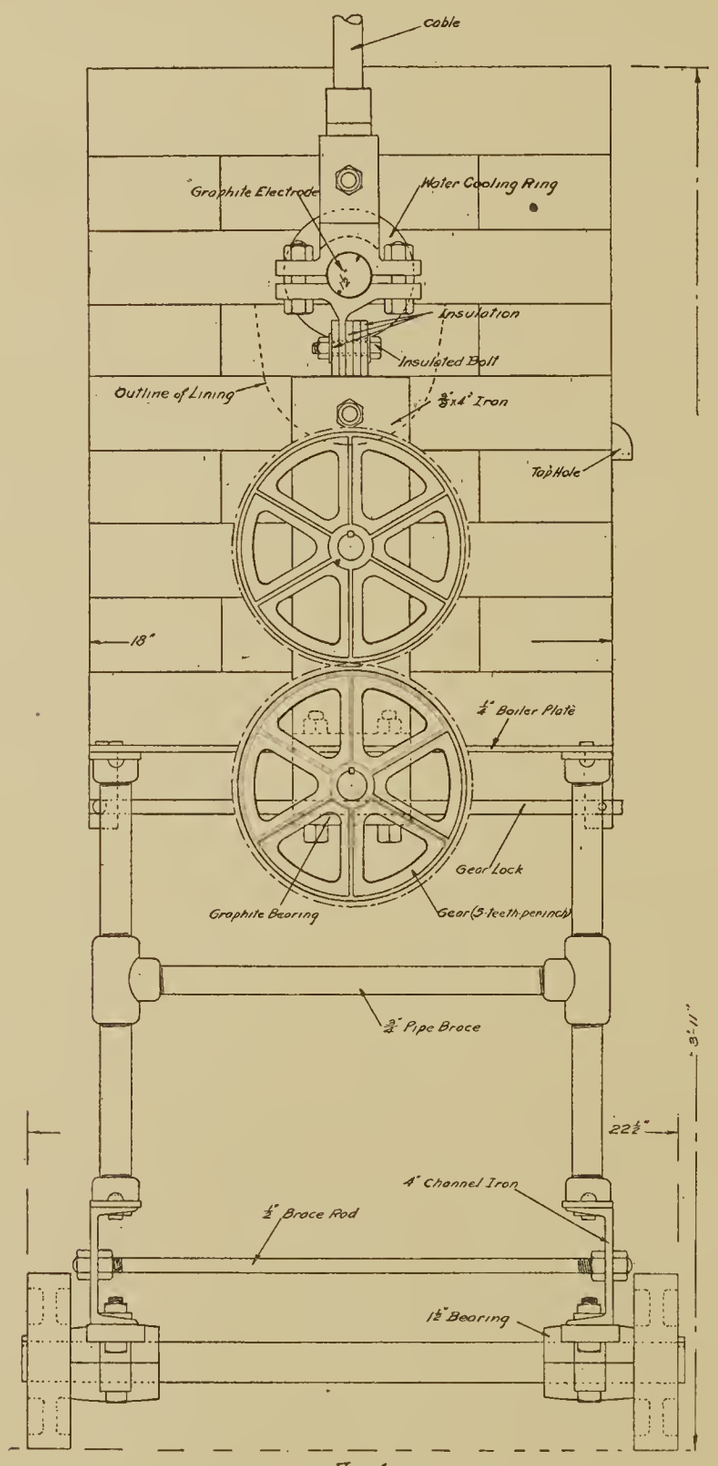

Fig 1

As shown in the drawing the furnace is built up so as to heat the charge in the crucible by radiation from the arc. In our work we generally use a current at 50 volts and 500 to 1,000 amperes.

The apparatus can also be used for other kinds of furnaces;

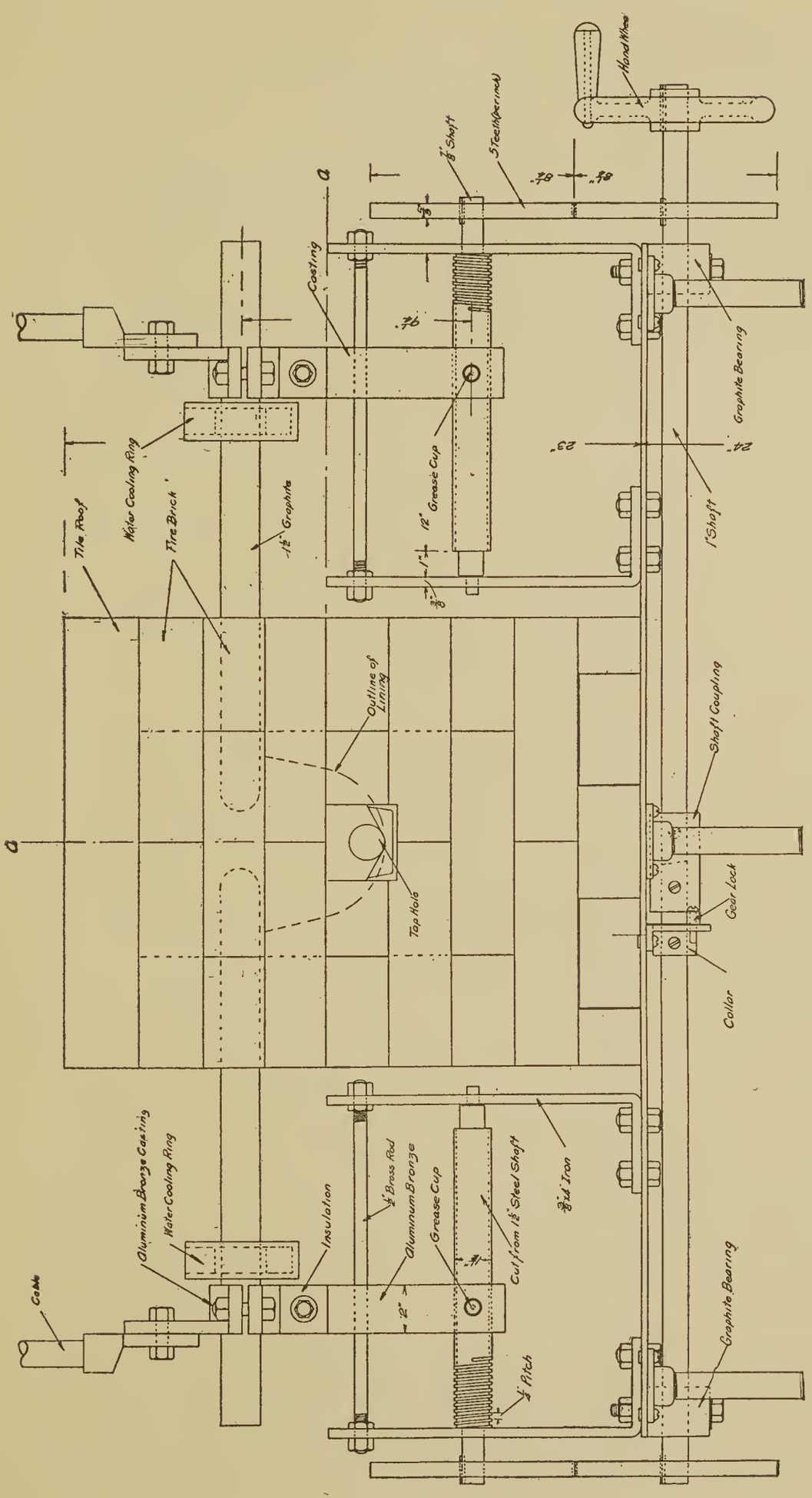
Hand Wheel
Steel Pinions
Casting
Graphite Bearing
Grease Cup
Water Cooling Ring
Fire Brick
Graphite
Tile Roof
Outline of Lining
Shaft Coupling
Tap Hole
Gear Lock
Collar
Aluminum Bronze Casing
Water Cooling Ring
Insulation
Brass Rod
Aluminum Bronze
Grease Cup
Cut from 1½ Steel Shaft
Cast Iron
Graphite Bearing
Pitch
Cable

FIG. 2.

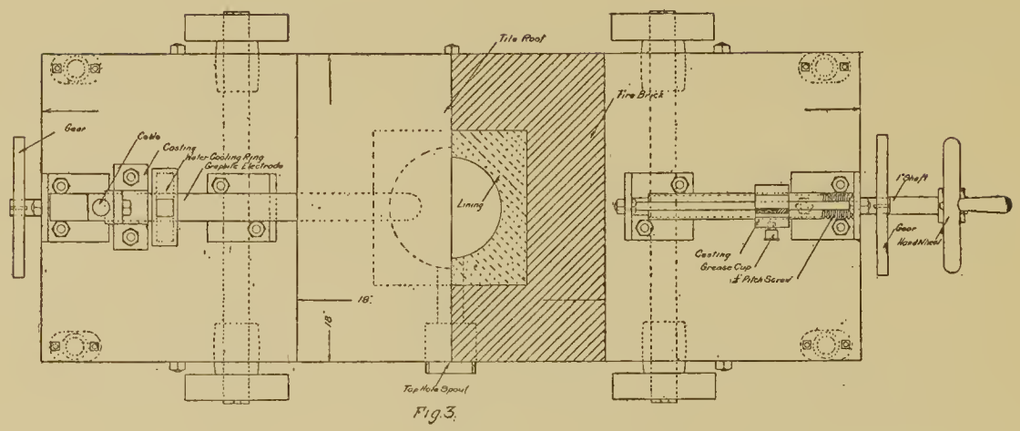

Fig. 3.

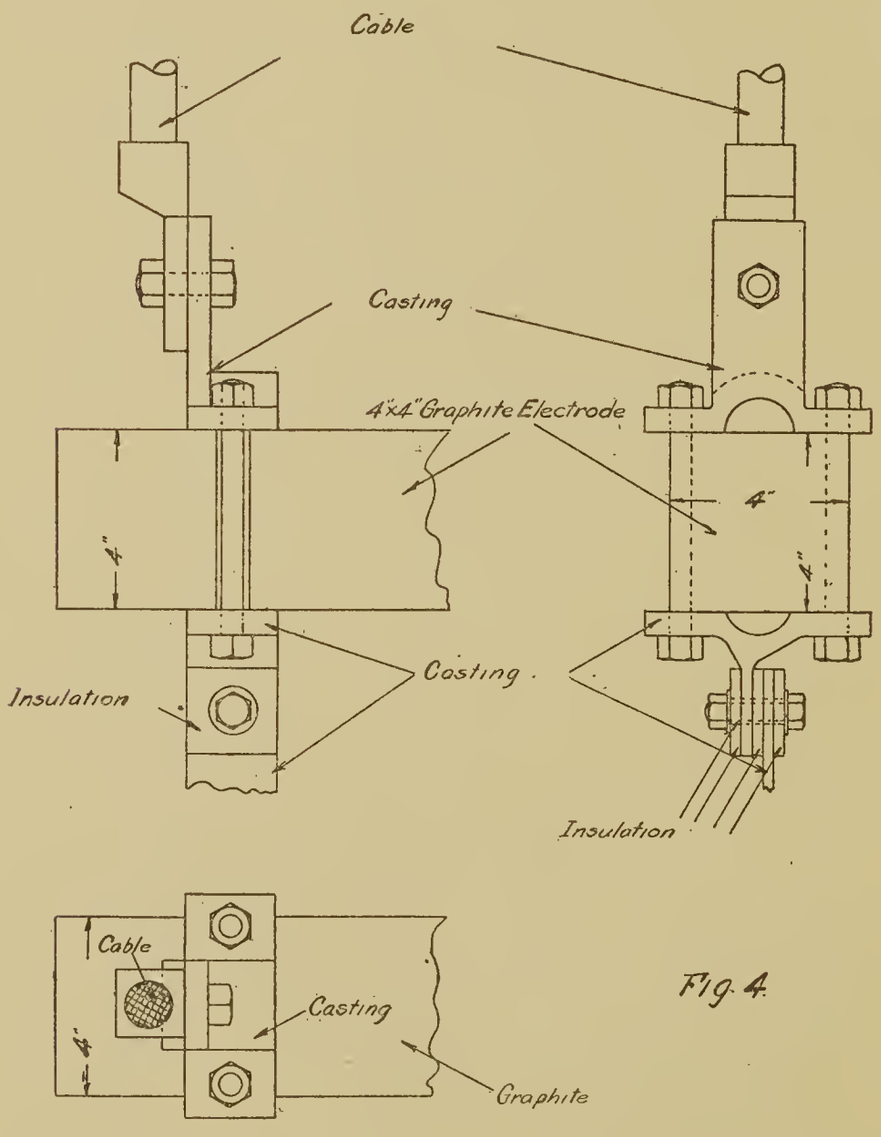

Fig. 4.

for example, for heating a crucible embedded in granular carbon. When used in this way convenient electrodes are 4 by 4 inches (10 x 10 cm.) graphitized carbons. Fig. 4 besides showing details of the electrode holder construction shows modification for use

with 4 by 4 inch carbons. The mobility of the electrodes permits of considerable variation in the resistance of the furnace while running with a granular carbon resistor.

The furnace also is convenient for heating the central portion of a horizontal tube embedded in a granular carbon resistor, and, of course, it may also be used as a furnace in which the tube is heated by an arc on the outside, but in this case the 1½ inch (3.8 cm.) electrodes are used.

While there is nothing original in the general principles of the furnace it is believed that the detail design may be found as useful by others as it has by us, on account of its being so cheap and easy to construct.

FitzGerald Laboratories,
Niagara Falls, N. Y.

A paper presented at the Thirty-sixth General Meeting of the American Electrochemical Society, in Chicago, September 24, 1919, President Bancroft in the Chair.

A SQUARE DEAL FOR THE ELECTRIC FURNACE.[1]

By H. G. Weidenthal.[2]

Abstract.

A polemic defending the electric furnace for steel-making against prejudices of the trade. The user is urged to acquaint himself thoroughly with electric furnace practice and furnish the best that can be produced by the electric furnace. Thus "he will do justice to himself and his business, and at the same time give the electric furnace a square deal."

The advantages of the electric furnace have been dwelt upon individually and as a whole by the proponents of the various types and makes of furnaces and to some extent the shortcomings of the electric furnace have been brought out and discussed. Among the users of electric furnaces the great majority are enthusiastic boosters, but it is true that a small minority have found that they made a mistake in chosing a type of equipment not suited to their particular class of work. In some instances the furnace was not sufficiently rugged to stand the strain so that the cost of upkeep plus the lost time of delays has discouraged the user.

Lack of practical melting and general steel plant experience has caused trouble for more than one high-class designer, making his first attempt on an electric furnace. Designers of steel plant equipment have learned by experience that simplicity and extreme strength are essential, because of the temperature conditions to be met and because of the mental attitude of men working around hot metal. When working in the heat and under the potential hazard prevailing around a furnace or mill, men are naturally less

[1] Manuscript received July 12, 1919.

[2] The James H. Herron Co., Cleveland, Ohio.

considerate of the welfare of machinery than men working under cooler and less hazardous conditions. Failure to recognize this on the part of some furnace builders has given grounds for criticism by old steel plant men. This is unfair to the electric furnace, for it can be designed sufficiently simple and rugged to meet steel plant requirements.

Another source for apparent dissatisfaction among users of electric furnaces is directly chargeable to the users themselves. There are two classes of offenders. First, those who are familiar with the steel or castings business but do not appreciate the exclusiveness of the new equipment which they install. Instead of employing the proper men to intelligently operate the furnace, they buy it as they would a new cupola or a ladle and turn it over to some old "dyed in the wool" melter or blower to abuse as he sees fit. If this man is not exceptionally progressive, he has a fear of electricity and may even entertain hallucinations as to the effect of the electric current on steel. At any rate he is seldom enthusiastic about his new care and does not give it a fair deal.

The second class of offender is the man or group of men who know little or nothing about the operating end of the steel business or castings business, whichever it may be, that they embark upon. They have heard that the electric furnace runs itself and is the panacea for all the ills of the steel business. They expect by the use of this great device to sidestep the long years of apprenticeship and honest endeavor that the steel man has gone through to produce good steel. To them, pouring practice, ingot practice, cogging, forging and rolling, with their many very important details of temperatures, time of heating, etc., are merely unimportant details, for as they think "the electric furnace makes steel that cannot go wrong." They may even conclude that the so-called technique of steel manufacture is all unnecessary, but what happens when they try to market their product? The steel is piped, full of blow-holes, seams, slag, and they reverse their ideas and blame it all on the furnace. Is this fair to the electric furnace?

Now at last we come to the user who is getting satisfactory results and is really pleased with his electric furnace. He is a booster for the make of furnace which he has purchased and gen-

erally speaking he is well disposed toward all electric furnaces. Apparently he is doing all he can for himself and the industry, but, is he really boosting the electric furnace to the unconvinced or partially convinced user of steel?

It is a proven fact that p r o p e r l y made electric steel is far superior to open-hearth steel, and that the best makes equal and in certain cases excel crucible steel. Electric tool steels have made their mark and are here to stay. High-speed steels, alloy tool and plain carbon tool steels are being made which compare well with the best crucible grades. Electric alloy die blocks are giving exceptional service and promise to put the cheaper grades off the market. Some automobile manufacturers and makers of automobile parts are specifying electric steel for drive shafts, gears, and in a few cases for drop forgings. This last field is still in its infancy, but there the need for high-grade steel is very urgent. A large number of failures in drop forgings are due to dirty steel and segregations found in the open-hearth forging steels.

Forge shops making machinery forgings, shear blades, blanking dies, etc., are beginning to appreciate the merits of electric alloy steels and are using them quite extensively.

In the steel casting business only very few plants are specializing in high-grade and alloy steel castings, although a very profitable field is being developed for alloy steel castings. The majority are competing with converter and open-hearth castings, both in price and with regard to quality. It is quite true that by using the electric furnace as a melting unit and attempting little or no refining, it is possible to produce steel for castings at the same cost or in some cases even cheaper than by the converter or small open-hearth. Another reason for the use of an electric furnace for castings is the flexibility and ease of analysis control.

It is perfectly legitimate for the casting manufacturer to take advantage of economies offered by the electric furnace, but if he does not make full use of the ability of the furnace to produce high quality steel, such castings should not be advertised as electric furnace product. The desire on the part of the manufacturer for large production and low cost should not become a reflection upon the electric furnace. It may be true that the trade does not demand a high-grade steel casting, but if "Electric Steel Cast-

ings" are sold to the trade, it should be supplied with material having a quality which is implied by the trade name "Electric Steel Castings."

Are the manufacturers of electric steel ingots, billets and bars producing the very best product that the furnace is capable of? Yes, some are producing excellent material, some are producing good material in the furnace but are not careful enough about their pouring, mould practice, cooling, reheating, forging or rolling. Others make good steel most of the time but feel that they cannot afford to remelt off heats, so they try to dispose of them either as their regular product or in some cases as open-hearth steel. Some of these heats are off in analysis as was the case with one lately brought to the writer's attention which analyzed 0.065 phosphorus but was sold as high-carbon chrome vanadium shear blade stock requiring tool steel quality. Of course the steel failed and went back to the producer after the purchaser had lost a great deal of time and money trying to use it. Another very recent example is of some 3.5 percent nickel steel for navy work. The analysis was correct but the steel was so full of non-metallic inclusions that it fell down miserably on its physical requirements. The writer is forced to admit that this steel was as bad or worse than any open-hearth steel he ever saw. It may be possible that in both of these instances the steel was made under adverse conditions such as bad bottom, bad roof or walls, forcing the operator to hurry the heat lest a breakdown catch him with his heat unfinished. This, however, is a poor excuse, for the proper course is that adopted by at least one plant which has a strict rule that no heat shall be rushed to completion because of furnace troubles, but that in such emergency the melter shall, as soon as possible, pour the heat w i t h o u t a l l o y s. This material can only be used for remelting, and there is not even a temptation to apply it on an order. At the same time the cost of alloys is saved.

In addition to furnace breakdowns, there are two other major causes for poor heats. One is the quick heat single slag method, that is, no dephosphorizing slag is removed. The result is not only higher phosphorus content, but all foreign substances adhering to the scrap remain in the furnace to contaminate the finishing slag. At times this practice makes it impossible to

obtain the proper conditions for finishing the heat so that the steel is no better and may be worse than the better makes of open-hearth steels.

The second cause has several angles and is not so very easily controlled. It is ignorance of proper temperatures, not only for pouring but for certain reactions which, as is not generally recognized, cannot occur except within certain temperature ranges. The common failing of pouring at too high a temperature is little worse than working a heat at too low a temperature. This temperature phase is too large a one to be included here but would be an excellent topic for a symposium.

An investigation of the two cases above and a number of other cases where electric steels have not lived up to their reputation, reveals that the makers of these steels are l o w b i d d e r s in each case, and the price was about that of open-hearth alloy steel. What does this indicate? It looks as if electric steel manufacturers were willing to sacrifice quality to compete in price with open-hearth steel. This is not all, some companies have gone still further, they offer "electric steel made to open-hearth specifications" at the price of open-hearth steel and another grade known as electric steel made to electric specifications at an advance of 2 cents per pound over the other price. Why should there be any call for such a policy?

The steel-consuming public has been educated to the need of higher grade steels during the war. The electric steel maker should continue to cultivate this good seed, instead of choking it off by prejudices born of this price-cutting policy. He should remember that when the price of open-hearth steel advanced from 250 percent to 400 percent, the price of his own product in a very few cases advanced as much as 100 percent in spite of the higher cost of power, labor and raw materials. Only a part of his raw materials have dropped in price, while labor and power are still advancing. Even during the artificial process of price-setting lately foisted upon the steel industry, no definite price has been set on electric quality steel and why should the manufacturers themselves start a campaign of cutting prices, and worst of all, cutting quality.

If the electric steel producer is an intelligent business man, he must needs realize that o n e t o n of carelessly made steel may

curtail more sales of electric steel than he could secure by cutting his price to below his cost. The public soon forgets the many good shipments he has made, but one bad lot of steel leaves a bad taste, which salesmanship of the higher caliber may slightly sweeten but can never completely eradicate.

An awful epidemic of poor steel has existed since the signing of the armistice, probably due to a letting down after war tension. This applies to all grades and makes of steel, but, as before mentioned, electric steel of late has not been all that it might be. Unfortunately, a large number of steel users are not equipped to thoroughly investigate a shipment of steel before putting it into work. They often spend more than the cost of the steel in forging, treating or machining before the fact is discovered that the material is defective. Of course the maker replaces the steel but he does not repay the user for the work he has done upon it nor for the delays due to replacement. A discriminating buyer could well afford to pay more for his steel if the maker was so sure of his product that he could guarantee not only replacement but guarantee the purchaser against any loss due to defective steel. Why not get the proper price and in turn give only the best that can be produced by the electric furnace.

Every man making electric steel, if he is a normal man, cannot help but develop a feeling of attachment for his furnace and for the industry. Let him do justice to himself and his business and at the same time give the electric furnace a square deal.

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